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OXIDATION-REDUCTION OF NATURAL GRAPHITE- A STEP TOWARDS SYNTHESIS OF GRAPHENE

L. M. Manocha*, Hasmukh Gajera, Vishal Mankadia and S. Manocha

Department of Materials Science, Sardar Patel University, Vallabh Vidyanagar

ABSTRACT

Carbon has been of great scientific and technological interest over all civilizations, mainly because of its application of its own but also in development of products through combinations with other elements. This interest is mainly due to the numerous structures it can be formed of. The latest interests are nanocarbons, mainly carbon nanotubes and graphene. Studies have been performed on wet oxidation of graphite, followed by exfoliation and reduction in order to synthesise graphene. Acid route has been followed for oxidation whereas reduction has been carried out in aqueous medium as well as in DMF. The reactions have been followed using FTIR, TGA and Raman spectroscopy. Reduction of exfoliated graphene oxide sheets in water with hydrazine results in a material with characteristics that are comparable to those of pristine graphite.

Key words: carbon, graphite carbon nanomaterials, graphene, graphite oxide.

INTRODUCTION

Carbon occupies very important place in the periodic table. Depending upon its electronic (sp , sp^2 or sp^3) configuration, it exists in various allotropic forms, amorphous carbons, two dimensional layered graphite or 3D networked diamond. All these forms possess distinct properties and applications, industrial to high tech. Applications again vary from monolithic to composites products with other elements. Therefore, there has been both scientific as well as commercial interest in this material as well as exploring new forms. The last two decades discoveries have been the Fullerenes and carbon nanotubes. Of all these, the planar or the so called graphitic structures with predominantly sp^2 bondings are well studied and also find many practical applications. All properties, mechanical, thermal and electrical conductivities of Graphite parallel to the sheets (graphene sheets) are greater than perpendicular to the sheets. However, the properties of the bulk graphite are marked by the stacking of the layers (Fig. 1). The bonds between atoms within a layer are very strong. However, the delocalized π orbitals make the forces between two layers of graphite very weak. Because of this, graphite layers can easily be shifted against each other or separated from each other to get individual graphene sheets. Till last decade, these graphene layers were thought to be thermodynamically unstable and hence getting individual graphene sheets were not explored. However, the current nanotechnology has generated interest in carbon nanomaterials. The existence of single wall carbon nanotubes evolved interest in exploration of single graphene sheets. Graphene is two-dimensional material constituting a new nanocarbon comprising layers of carbon atoms arranged in six-member rings. It is distinctly different from carbon nanotubes (CNTs) and fullerenes, and exhibits unique properties which have fascinated the scientific community.

As also shown in Fig. 1, it can be wrapped up into 0D fullerenes, rolled into 1D nanotubes or stacked into 3D graphite [1]. Graphene, promises a diverse range of applications from composite materials to quantum dots [2-4]. Graphene and chemically modified graphene (CMG) are promising candidates as components in applications such as energy-storage materials, 'paper-like' materials [8] polymer

composites, liquid crystal devices and mechanical resonators, field-effect transistors (FET), supercapacitor electrode applications [7], hydrogen storage and biomedical applications, due to its excellent properties [3-5]. Ideally graphene is a single-layer material, but graphene samples with two or more layers are being investigated with equal interest.

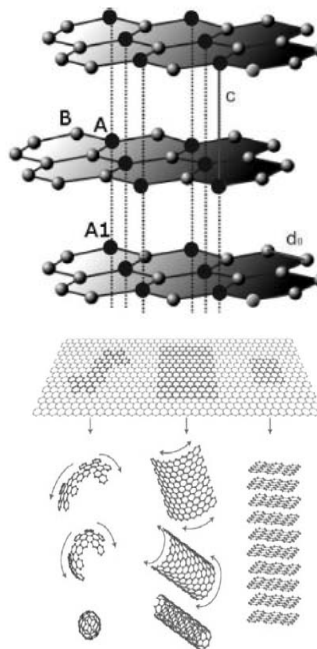


Fig. 1 Schematic of a graphite structure consisting of three graphene layers, and Graphene: the parent of all graphitic forms, fullerenes, carbon nanotubes and graphene.

Although single-layer graphene and bilayer graphene were first obtained by micro-mechanical cleavage in 2004 at the Centre for Mesoscopic and Nanotechnology of the University of Manchester, UK, directed by A K Geim [1]. Graphene was initially isolated by mechanical exfoliation, peeling off the top surface of small mesas of pyrolytic graphite [3-5], a method which is not suitable for synthesis of single-layer graphene or of few-layer graphene (FG). Among other methods and procedures for large-scale synthesis, exfoliation of natural

*Corresponding author: manocha52@rediffmail.com

graphite is the most convenient technique. The natural graphite could be as such or that containing any oxygen functionalities. Latter involves the exfoliation of graphite oxide (GO) followed by reduction. Several methods have been described in the literature for preparation of oxidized graphite or so called graphite oxide [6] Graphene and Chemical modification of graphene oxide, which is generated from graphite oxide (GO) has been a promising route to achieve mass production of CMG platelets [7,8]. Though GO has a history that extends back many decades to some of the earliest studies involving the chemistry of graphite, the interest in oxidation of graphite and its reduction is regenerated due its versatility in production of graphene in large scale at comparatively low price. Present studies were performed to evolve methods wherein the natural graphite flakes have been chemically treated followed exfoliation to yield grapheme [7,9]. All these studies have been followed using spectroscopic techniques and thermal analysis.

MATERIALS AND METHODS

Materials

The natural graphite flakes (particle size – 400 μm) was used as the starting material for preparation of graphene. For this process other chemical reagents used were sulfuric acid (H_2SO_4) (Ranbaxy fine chemical), Potassium persulphate ($\text{K}_2\text{S}_2\text{O}_8$) (Loba chemicals) Phosphorus pentoxide (P_2O_5) (Sigma – Aldrich,), Potassium Permanganate (KMnO_4) (Samir Tech chem.), Hydrogen peroxide (H_2O_2) (RFCL), Acetone (Qualingens fine chem), Dimethyl Formamide (DMF) (Qualingens), Hydrazine Hydrate ($\text{H}_6\text{N}_2\text{O}$) (RFCL).

Synthesis of Graphene

The preparation of graphene involves three key steps.

- Oxidation of starting material (Graphite flakes) to synthesize Graphite Oxide (GO).
- Thermal expansion or Exfoliation of the as – prepared Go to obtain TEGO/EGO.
- Reduction and dispersion of resulting EGO to produced graphene.

Synthesis of graphite Oxide (GO)

The employed method consists of two steps. (i) Pre Oxidation of Graphite, (ii) Oxidation of POG (Pre – Oxidized Graphite).

Sulfuric acid (H_2SO_4), potassium persulphate ($\text{K}_2\text{S}_2\text{O}_8$) and phosphorus pentoxide (H_2O_5) were mixed and stirred at room temperature till clear solution was obtained. In this mixture $\text{K}_2\text{S}_2\text{O}_8$ and P_2O_5 were added slowly and stirred till clear solution was obtained, Graphite flakes were added slowly in this mixture. After this addition, mixture was stirred for 4 – 5 hrs at 80°C . After stirring for 4 – 5 hrs, dilution of this solution was carried out. For this process distilled water was slowly added to above solution keeping temperature below 10°C . Reaction flask was immersed in ice bath for dissipation of heat in exothermic reaction that occurred during addition of water. Finally, the resulting suspension was filtered and washed with distilled water up to neutral pH and dried it in oven at 100°C for 1 – 1.5 h to obtain Pre – Oxidized Graphite (POG).

Oxidation of POG

Sulfuric acid and potassium permanganate were mixed and stirred at a 10°C for half an hour. Pre – Oxidized Graphite (POG) was added slowly and stirred for 1 hour in ice bath followed by stirring at room temperature for 24 h. Distilled

water was added to the solution. Reaction flask was immersed in ice bath to control the temperature below 10°C . An exothermic reaction occurred during addition of water. This solution was treated with H_2O_2 to reduce residual permanganate to soluble manganese ions. The solution became bright yellow colored after reduction. After this process, this suspended solution was washed with distilled water by centrifugation decantation till neutral pH obtained. Finally the resulting suspension Graphite Oxide slurry (GO) was obtained.

Exfoliation of Graphite Oxide

The as prepared GO was exfoliated using ultrasonication method to synthesize Exfoliated Graphite Oxide (EGO) or Graphene Oxide.

Thermal Expansion of Graphite Oxide

The Graphite oxide was thermally expanded to form TEGO by rapidly heating it in a furnace shown in Fig. 2. The nitrogen gas was flushed in the quartz tube containing graphite oxide which was then rapidly moved in to the middle heating zone of the furnace already heated to 220°C .

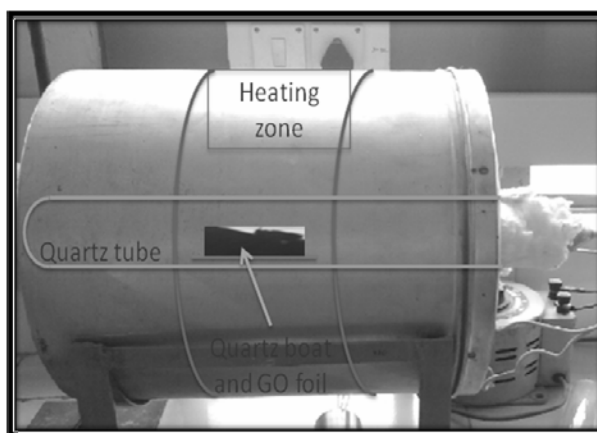


Fig. 2 Experimental setup for synthesis of TEGO

Reduction and Dispersion of Graphene Oxide

Reduction of Graphene Oxide in Water

In typical procedure GO slurry was loaded in a round bottom flask to which was added distilled water yielding an inhomogeneous yellow solution. This solution was sonicated by using ultrasonic bath cleaner until it became clear without any visible particulate matter in solution (45 min – 1 h). To obtain homogeneous dispersion of exfoliated graphite oxide (Graphene Oxide) in water Hydrazine Hydrate was added to this solution and solution was heated at 100°C in paraffin bath for 24 h. The reduced GO (r - GO) gradually precipitated out as a black solid on the surface. This product was isolated by filtration over a medium fritted glass funnel. It was washed with distilled water (10×150 ml) up to neutral pH and dried at 100°C in oven for 3 h.

Reduction of GO in DMF

In this experiment, Graphite Oxide (30 mg) was loaded in reaction flask containing 10 ml water. This solution was sonicated for 1 h to obtained homogeneous dispersion of exfoliated GO (Graphene Oxide). Addition of Dimethyl Formamide (DMF, 90 ml, volume ratio DMF/ H_2O = 9) yielded the light – brown suspension of graphene oxide (EGO). The suspension was quite stable. To the graphite Oxide suspension,

Hydrazine Hydrate was added and stirring was done on magnetic stirrer by using Teflon coated magnetic bar for 12 h at 80°C. Finally, the reduced graphene oxide was obtained as black colloidal suspension in Dimethyl Formamide (DMF).

Characterization of GO and r-GO

Electrical Conductivity measurement of GO and r-GO

The electrical conductivity of graphite flakes, Graphite Oxide, Reduced Graphite oxide was measured by Keithley – 6182 AC/DC current source – Nanovoltmeter (Fig. 3). In this experiment samples were used in powder form.

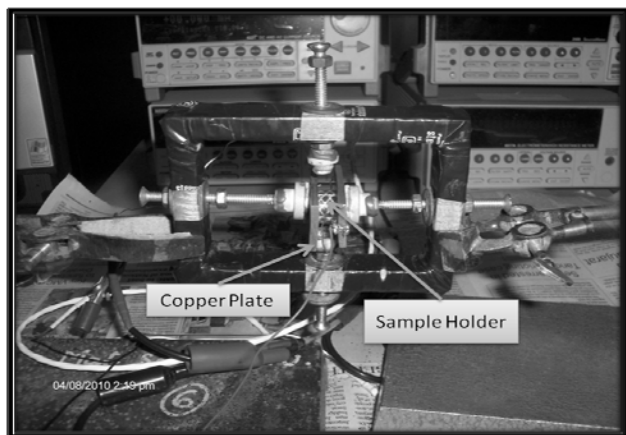


Fig. 3 Sample holder and set up for measurement of electrical conductivity

Thermogravimetric study

Thermogravimetric analysis (TGA) is performed on samples to follow oxidation reaction, to determine the extent of oxidation of graphite, reduction process etc. TGA was done from room temperature to 800°C at a heating rate of 5°C per min.

Fourier Transform Infrared Spectroscopy (FT – IR) Study

SHIMDZU 8300 FTIR spectrometer was used to study the type of chemical groups present in GO and r-GO.

Surface area measurement

The surface characterization, e. g. surface area, pore size, pore size distribution were determined by using BET apparatus 2375 Gemini Micromeritics. The nitrogen gas adsorption measurement was carried out at liquid nitrogen temperature.

Raman spectroscopic study

Raman scattering is a fast and non destructive technique that provides a direct insight on the electron – phonon interactions, which implies a high sensitivity to electronic and crystallographic structures. It has been extensively applied to the structural investigation of carbon materials such as carbon nanotubes and graphene. Raman spectra of graphite and its compounds were taken on Renishaw Raman microscope using 514 laser as excitation source.

RESULTS AND DISCUSSION

Exfoliation of GO in water

Exfoliation of GO in water showed the hydrophilic nature of the oxygenated graphene layers and its easy exfoliation in aqueous media. As a result, GO readily formed stable colloidal

suspensions in water. These sheets are, however, different from graphitic nanoplatelets or pristine graphene sheets due to their low electrical conductivity. Moreover, the resulting sheets, by TEM analysis, were found to be about 1 nm thick leading to a conclusion that complete exfoliation of GO to individual graphene oxide sheets was achieved under these conditions. These are different than pure graphene sheets which are smaller in thickness. Graphene oxide sheets are expected to be ‘thicker’ due to the presence of covalently bound oxygen and the displacement of the sp^3 hybridized carbon atoms slightly above and below the original graphene plane.

Reduction of exfoliated GO

During the hydrazine reduction of graphene oxide sheets dispersed in water, the brown-colored dispersion turned black and the reduced sheets aggregate and eventually precipitate. The precipitation of the reduced sheets occurred, presumably due to their becoming less hydrophilic as a result of oxygen removal and thus increased incompatibility with the aqueous medium. As dissolution of the reduced sheets decreases, the inter-sheet hydrophobic interactions cause them to aggregate as well as adhere to the hydrophobic surface. The main purpose of reduction of EGO was to obtain graphene from the graphene oxide. Hydrazine is known to open epoxide rings readily and to form hydrazino alcohols. Though, such a reaction pathway is possible in GO upon hydrazine treatment, there are other complementary reactions resulting in oxygen removal. It is possible, however, that the initial derivative produced by the epoxide opening with hydrazine reacts further via the formation of an aminoaziridine moiety which would then undergo thermal elimination of diimide to form a double bond. Such a reaction might be further driven in GO by re-establishment of the conjugated graphene network.

Electrical conductivity studies of as such graphite, GO and r-GO

To determine the extent to which the chemical reduction of exfoliated GO restores the electrical properties of the graphitic network, the measurement of electrical conductivity of compressed-powder samples of the pristine graphite, GO, and the reduced GO at room-temperature were made. In this study samples were used in the powder form. The electrical conductivity was found to decrease after oxidation but an increase in the conductivity was found after reduction. The conductivity of the r-GO was more than that of the GO because of the restoration of the sp^2 network of the graphitic plane by reduction of GO. The value of the electrical conductivity of the graphite flake, GO and r-GO are $0.6713 \Omega^{-1} m^{-1}$, $0.0040 \Omega^{-1} m^{-1}$ and $0.0245 \Omega^{-1} m^{-1}$ respectively.

Raman spectroscopic study

A typical Raman spectrum of Graphite flakes, Pre – Oxidized Graphite and Graphite Oxide is shown in Fig. 3. The significant structural changes occurring during the chemical processing from pristine graphite to GO, and then to the reduced GO, are also reflected in their Raman spectra. The Raman spectrum of the pristine graphite, as expected, displays a prominent G peak as the feature at $1584 cm^{-1}$ and at $2656 cm^{-1}$, historically named G', since it is the second most prominent peak always observed in graphite samples. In the Raman spectrum of GO, the G band is broadened and is found to be shifted to $1604.8 cm^{-1}$. In addition, the D band at $1373.28 cm^{-1}$ becomes prominent, indicating the reduction in size of the in-plane sp^2 domains, possibly due to the extensive oxidation. The Raman spectrum of the Pre – Oxidized Graphite (POG) also contains both G and D bands at 1584 and $1317 cm^{-1}$ respectively with an increased D/G intensity ratio in GO compared to that in the POG spectrum. This change suggests a decrease in the average size of the sp^2 domains in GO.

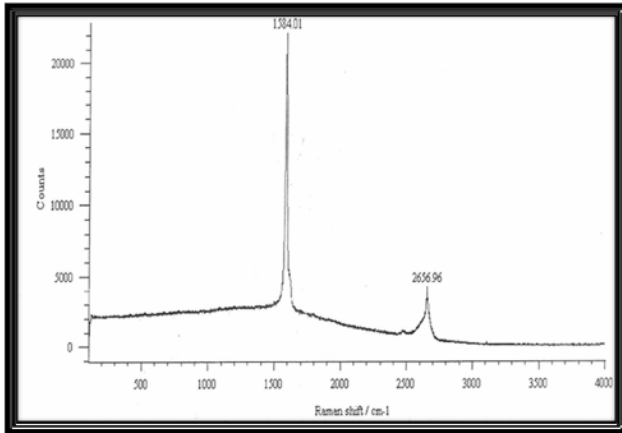


Fig. 3a

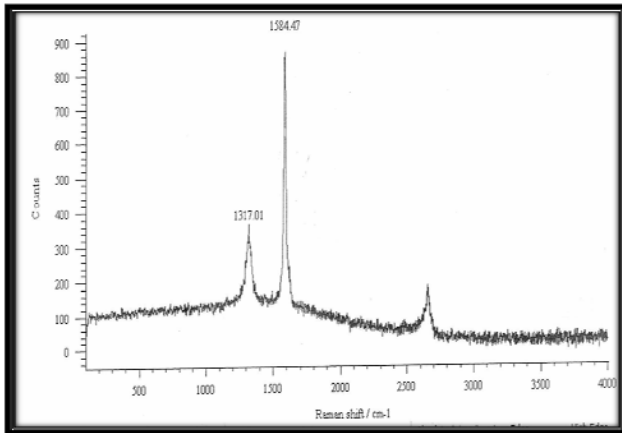


Fig. 3b

TGA studies of GO and r – GO

GO is thermally unstable and starts to loose mass upon heating even below 100° C, the major mass loss occurs at near 220° C, presumably due to pyrolysis of the labile oxygen-containing functional groups, yielding CO, CO₂, as seen in Fig. 4. Hence, the thermal decomposition of GO can be accompanied by a vigorous release of gas, resulting in a rapid thermal expansion of the material. This is evident by both large volume expansion and a larger mass loss (from flying GO debris in the TGA instrument) during a more rapid heating regime.

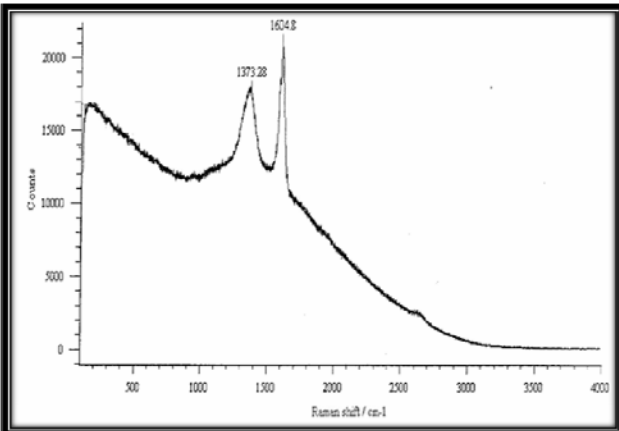


Fig. 3c

Fig. 3 (a, b, c) Raman spectrum of (a) Graphite (b) Pre-oxide Graphite (PGO) and (c) Graphite oxide (GO).

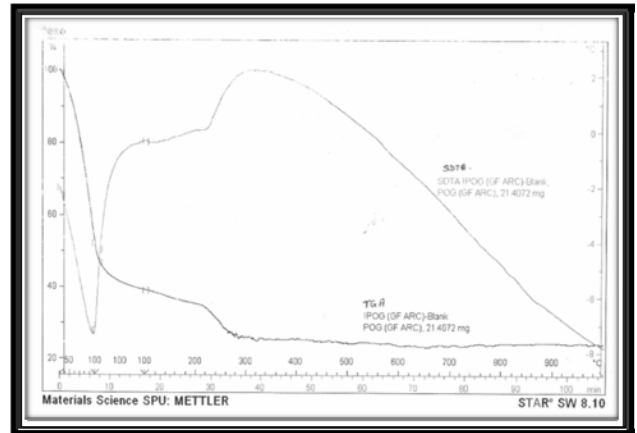


Fig. 4 (a).TGA spectrum of Pre - Oxidized Graphite (POG)

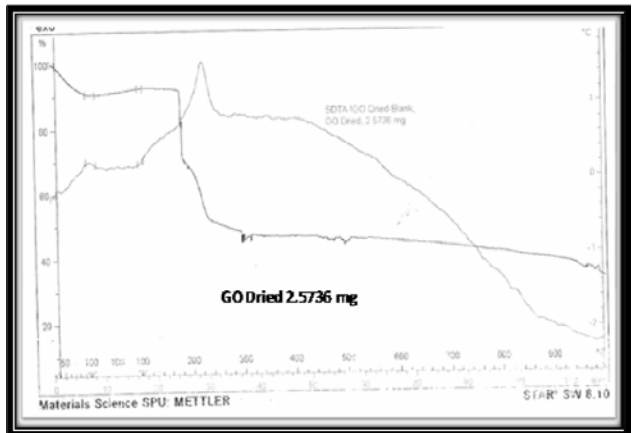


Fig. 4 (b) TGA spectrum of Graphite Oxide (GO)

On the other hand, the removal of the thermally labile oxygen functional groups by chemical reduction results in much increased thermal stability for the reduced GO. Apart from a slight mass loss below 100° C, which can be attributed to the loss of adsorbed water, no significant mass loss is detected when this material is heated up to 600° C. TGA curve of Pre – Oxidized Graphite and graphene oxide exhibits peak at 230° C, which correspond to 10% and 30 % weight losses respectively. These peaks are attributed to the removal of oxygen containing groups (second peak). But in case of the r – GO, there was very small peak observed at 220°C because of the removal of oxygen containing groups from the Graphene oxide layer. It means that during the reduction of Graphene Oxide large amount of oxygen containing functional groups are removed, but there were some groups still present on graphene oxide plane.

FT – IR Study of GO and r – GO

An additional tool for the characterization of graphene oxide is FTIR spectroscopy. Fig. 5 shows FTIR spectra of graphite, GO and r-GO. Graphene oxide exhibits the following characteristic IR features. Strong band observed at ~ 3440 cm⁻¹ attributed to the hydroxyl stretching vibrations of the C-OH groups, doublet near ~2925 cm⁻¹ and ~2855 cm⁻¹ attributed to the CH₂ or CH₃ groups, a weak peak in between 1610 - 1627 cm⁻¹ assigned to the C=O stretching vibrations of the-COOH groups, a weak peak at 1396 cm⁻¹ assigned to the O-H deformations of the C-OH groups, and a strong band at 1050 - 1095 cm⁻¹ attributed to C-O stretching vibrations. FT – IR

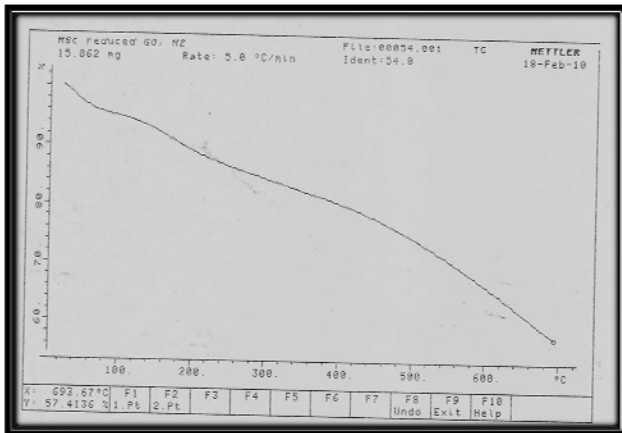


Fig. 4 (c) TGA spectrum of reduced Graphite Oxide (r - GO)

spectrums of Graphite flakes, POG, GO, r - GO at room temperature and r - GO heated at 100° C as shown in below figures respectively. Fig. 5 shows FTIR spectra of graphite, GO, POG and r-GO. The FTIR spectra of POG and GO show band corresponding to C=O stretching vibration of the -COOH group stronger than for graphite because of the oxidation reaction of graphite which generate acidic functional groups. The -OH band of POG and GO sample are also stronger and broader than for the GF sample and a weak band at 1396 cm⁻¹ assigned to the O-H deformations of the C-OH groups was observed in POG and GO sample. In case of reduced graphene oxide, a weak shoulder at 3430 - 3440 cm⁻¹ attributed to the hydroxyl stretching vibrations of the C-OH groups was absent and a weak band at 1396 cm⁻¹ assigned to the O-H deformations of the C-OH groups was also absent. A weak band at 1610 - 1627 cm⁻¹ assigned to the C=O stretching vibrations of the-COOH groups disappeared in the r-GO spectrum. Absence of these groups in r-GO sample are because reduction reaction between hydrazine hydrate and graphene oxide so that these groups get removed from the graphene oxide. But after reduction some new functional groups were attached to the graphene plane such as -NH₂ groups at 1541 cm⁻¹ and -C=N group at 1681.8 cm⁻¹.

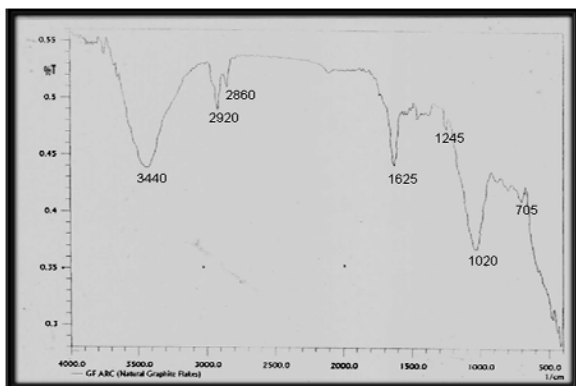


Fig. 5a

Surface area study

Surface area measurement of the reduced GO sheets and exfoliated GO sheets via nitrogen gas absorption yielded a surface area value of 382 m²/g, 466 m²/g respectively. This high specific surface area is partially an indication of the degree of GO exfoliation. However, it is still lower than the theoretical specific surface area for completely exfoliated and isolated

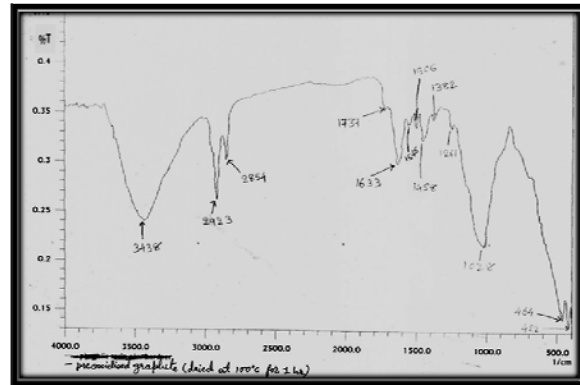


Fig. 5b (POG)

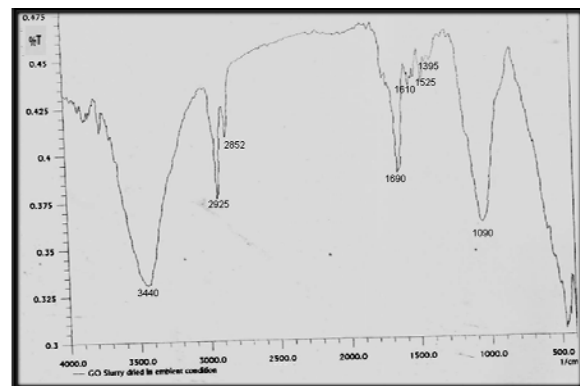


Fig. 5c

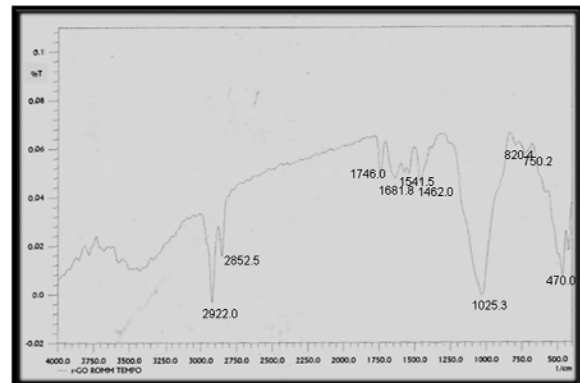


Fig. 5d

Fig. 5 FT - IR spectrum of (a) graphite, (b) POG, (c) GO and (d) r-GO

graphene sheets (2,620 m²/g), But after reduction of Graphene oxide the surface area of that r - Go was 415 m²/g this is due to the agglomeration of the graphene oxide sheets upon reduction. While this agglomeration can result in the partial overlapping and coalescing of the reduced sheets and lowering the surface area of the bulk materials, the crumpled 3-dimensional structure of the sheets still leaves many exposed surfaces.

CONCLUSION

The graphene sheets have been synthesized by a fast method through oxidation-reduction technique using graphite flakes as a starting material.

Reduction of exfoliated graphene oxide sheets in water with hydrazine results in a material with characteristics that are comparable to those of pristine graphite.

The characterization of the reduced GO indicates that the hydrazine treatment results in the formation of unsaturated and conjugated carbon atoms, which in turn imparts electrical conductivity.

As such, reduced graphene oxide sheets may find use in a variety of applications such as hydrogen storage and as an electrically conductive filler material in composites.

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