Thermo Gravimetric Analysis and FTIR Analysis of Electrochemically Synthesized Graphene Oxide (GO)/Reduced Graphene Oxide (rGO)

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Abstract. Excellent mechanical, electrical, thermal and optical properties have been demonstrated by Graphene, a monolayer type of carbon with two dimensional honeycomb lattices (sp2 hybridization). For many applications in the fields of electronics, bio-medicine, composites, sensors, actuators and energy storage and conversion devices, it is a promising part. There are two methods (top down method and bottom up method) for the synthesis of the graphene oxide (GO)/reduced graphene graphene oxide (rGO). In the present work the objectives are to investigate the thermal stability and FTIR analysis of the graphene Oxide (GO)/reduced graphene Oxide (rGO). The material is thermally reduced at 650°C for the 4 hours at a heating rate of 20°C/min. FTIR shows the presence of various chemical compounds inside the sample.

1. Introduction
The graphene, which is a two-dimensional hexagonal honeycomb like structure, has attracted great deal of attention: the one-atom dense graphitic, sp2-bonded carbon content. This material has numerous new interesting properties, such as intrinsically superior electrical conductivity (106 Ω-1 cm-1), almost transparent in visible light (97.7%) [1], relatively high carrier mobility (2.45×105 cm2 V-1 s-1) [2], with high specific surface area (2635 m2 g-1) [3], superior mechanical stability (Young’s modulus ≥ 1.1 TPa) [4], and high thermal conductivity (above 3500 W mK-1) [5]. These characteristics that make graphene one of the most promising upcoming materials that may have applications in electronics, photonics and many other fields. The biggest challenge in the field of organic electronics is to create an electrode that, apart from being rather durable, should be highly conducting, versatile and transparent[6]. Graphene synthesis has been explored via different routes, such as micromechanical cleavage. CVD, the process of thermal vapour deposition, etc. This takes time and the yield is low. Electrochemical synthesis is considered as a cheaper and greener process that is theoretically capable of mass production of few-layer graphene (FLG). Using the electrochemical exfoliation approach, FLG off graphite at the anode in ionic liquids [7, 8].

This solution-based technique enables the processing of graphene. This solution-based approach makes graphene suitable for film-shaped processing on most substrates, including flexible forms. A new one-step process has been developed recently for the synthesis of high-quality FLG. In the year 2011, this approach was first reported by Ching-Yuan Su et al. Electrochemical graphite exfoliation in
a 0.5 M H2SO4 solution using graphite as anode and platinum as cathode was performed in this process. High-quality graphene but with a lower yield (up to 8 percent wt) was obtained. The graphene sheets thus obtained were processed and transformed via dimethylformamide (DMF) into a film like structure incorporating several nearby graphene sheets. Recently, Parvez et al have been using the electrochemical exfoliation method for graphene synthesis. They obtained comparatively large graphene sheets (lateral scale ~10 μm) by tuning the H2SO4 solution acidity to 0.1 M solution [7]. Therefore, it is clear that electrolytes play a crucial role in the yield and efficiency of graphene synthesized electrochemically.

2. Experimental Section

2.1 Materials used

(i) Graphite flakes (10B grade) (ii) 1M Sulphuric Acid (H2SO4 98.08%) (iii) Diluted HCL (iv) Deionized water (HM2 Filter paper) (v) H2O2

2.2 Synthesis of Graphene Oxide (GO)/Reduced Oxide

Electrochemical exfoliation was conducted in a sulphuric acid aqueous solution as shown in fig. 1 (a) and (b). The electrolytic cell has graphite rod (in cylindrical form) as an anode and cathode. The diameter of the anode and cathode was taken approx 5mm. The anode was connected with the +ve (positive charge) and the cathode was connected with the –ve (negative charge). The gap between anode and cathode was maintained about 50mm. The anode and cathode both was immersed in the electrolytic solution about 50% of its length but should not touch the surface of the beaker. To prepare electrolytic solution, the sulphuric acid Fig. 2 (Thermo Fisher Scientific: 98.02%) is dissolved in the DI water (deionized Water). For the electrochemical synthesis of graphene 1M of dissolved sulphuric acid has been used. The ECE (electrochemical exfoliation) experiment has been conducted by applying direct current voltage of +15 V to the anode upto the end of the experiment. The application of high voltages to anode lead towards progressive graphite exfoliation through the surfaces. There are two different types of graphitic flakes formed during the exfoliation; one is sedimented at the bottom, composed of large graphitic pieces. The second form of graphitic sample floats on the electrolyte surface. These flakes are just about transparent yet only a few surfaces of graphene are found to consist of few layer of graphene. After the exfoliation the material is filtered with the help of filter paper fig. 3 (HM 2 grade with 12.5 cm diameter). After filtration the exfoliated graphite oxide was collected in a borosil beaker as shown in the fig. 4. Then the exfoliated graphite oxide is heated inside the core oven as shown in the fig. 5 to remove the excess moisture from the graphite oxide [9-13].

2.3 Thermal Reduction of GO

There are several techniques for the reduction of the Graphene Oxide (GO) like: green reduction, thermal reduction, acid and base reaction, etc. In this experiment the thermal reduction process were used to produce the reduced oxide of graphene (rGO) [14] [15]. For the thermal reduction muffle furnace were used as shown in the fig. 6. The obtained graphite oxide were heated/reduced inside the muffle furnace at a fixed temperature of 650°C for 4 hours with heating rate of 20 °C/min. The collected samples were named as EX1ATRGO650.
3. Results and Discussion

3.1 Thermal analysis
Thermogravimetric analyses of the samples were conducted using a DSC-TGA thermal analyzer (Mettler Toledo Star* System) instrument (as shown in figure no. 7) with a heating rate of 20 °C /
min in the nitrogen environment, from 5 °C to 800 °C. To estimate the thermal stability of the prepared sample, TGA was used. The parameters were selected for the analysis was: the initial weight of the sample was taken 6.2 mg, heat flow rate 2749.1999 mW. The sample temperature 335.82 °C, cell temperature 310.94 °C were observed after 17 minutes.

TGA curve of graphene oxide (GO)/reduced graphene oxide (rGO) of the sample as shown in Fig. 8. TGA curves showed that the total weight loss of graphene oxide (GO)/reduced graphene oxide (rGO) was about 32.00%. The loss of weight approx 3.25% up to 100 °C can be attributed to the evaporation of the moisture content and interstitial water, while the loss from 100 °C and upto 500 °C the total weight loss of the sample was observed about 6.4%. , finally the rapid weight loss 25.6% were observed from 500 °C and upto to 800 °C [16-18].

3.2 Fourier Transform Infrared Spectrometry (FTIR)

The FT-IR studies was followed by the methodology introduced by (Jagmohan, 2005) [5]. The lyoephylized resin controlled samples have been blended with dry bromide potassium pellet (KBr) and exposed to a pressure of approximately 5x106 Pa in ano create a clear reflective disc of an evacuated die of 13 mm in diameter and 1 mm thick. Area 40000 IR Spectra- at room temperature, 400 cm\(^{-1}\) was reported on a Perkin-Elmer Fourier spectrometer for transformation fitted with air-cooled DTGs Detecto (deuterated triglycine sulphate)

Figure 9 represents the FTIR spectra of the thermally reduced graphene oxide (GO)/reduced graphene oxide (rGO) [14, 15]. Many oxygen functional groups [14] were introduced into the structure of graphene oxide (GO)/reduced graphene oxide (rGO) when graphite was oxidised, which has a large peak at about 4000 cm\(^{-1}\) to 3600 3421 cm\(^{-1}\)represents reflecting stretching vibrations of (-OH) resulting from adsorbed water. The peak observed around 2400 cm\(^{-1}\) due to (O=C=O) stretching of carbon dioxide (CO\(_2\)) & shows the presence of carbon dioxide (CO\(_2\)) in the sample, the peak observed around between 1800 cm\(^{-1}\) to 1650 cm\(^{-1}\) due to (C=O) stretching of primary amide, the another peak observed around between 1650 cm\(^{-1}\) to 1400 cm\(^{-1}\) due to (C=C) stretching of alkene and shows the presence of methyl group, the peak observed around 1400 cm\(^{-1}\) to 900 cm\(^{-1}\) very broad and strong due to (S=O) stretching of the sulfonyl chloride, the peak observed around 1100 cm\(^{-1}\) very broad and strong due to (C-O) stretching of the tertiary alcohol, the peak observed around 1000 cm\(^{-1}\) to 800 cm\(^{-1}\) broad and strong due to (C=C) bending of the alkene and shows the presence of the vinylidene group [19, 20].

![Thermogravity Analysis Curve](image)

**Fig. 8** TGA curve of the graphene oxide (GO)/reduced graphene oxide (rGO)
4. Conclusions:

The consequences of rising reduction time on the microstructural properties, surface area of the layer, thermal stability and chemical presence of the sample was investigated in the study. In the TGA analysis total weight loss from 0°C up to 800°C was 32.00%. The FTIR analysis shows the presence of various functional groups like: carbon dioxide, alkene, methyl group, sulfonyl chloride, tertiary alcohol and vinylidene group.

5. References

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Fig. 9 FTIR spectra of graphene oxide (GO)/reduced graphene oxide (rGO)
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