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Graphene Oxide Fabrication Using Modified Hummer's Approach, then its Chemical Reduction to Obtain Reduced Graphene Oxide

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Abstract

This research describes how to synthesize graphene oxide following modified Hummer's technique, then its chemical reduction to produce reduced graphene oxide as a final graphene-like product. Due to its peculiar properties, graphene, a distinct carbon allotrope with an atom-thick structure, is regarded as a mysterious substance in the modern day. Because of its incredible electronic, mechanical, optical and thermal characteristics, it has attracted attention of researchers on a global scale. Recent developments have shown that graphene-based materials can significantly improve the properties of chemical sensors, nanocomposites, electrical and electronic equipment, optoelectronics, and energy storage devices. This study focuses on a modified synthesis of Graphene Oxide (GO), a crucial building block in the chemistry of graphene, and transforms graphene oxide back into reduced Graphene Oxide, a structure resembling 2D graphene. Electrochemical methods, XRD, and FTIR examinations provided proof that reduced graphene oxide was produced from graphene oxide, which was initially obtained from powder of graphite.

Subject Areas

Materials Science, Electrochemistry, Nanomaterials

Keywords

Graphene Oxide, Characterization, Spectroscopy, Reduced Graphene Oxide

1. Introduction

Starting from its first introduction, the two-dimensional material graphene has been used for a wide range of scientific as well as technological sectors for a variety

of purposes, such as sensors, electronics, energy conversion and storage, aerospace applications, biomedical engineering, etc. [1]. Some of the identified characteristics of the idealized graphene or pure infinite two-dimensional sheet of hexagonal carbon structure, including a high carrier mobility, large specific surface area, and decent mechanical properties, are listed in many existing reports as a deciding factor for graphene applications [2]. However, as pristine graphene is almost nonexistent, this can be viewed as an exaggeration. In addition, hypothetical graphene would also be impractical in technology areas such as energy storage and conversion [3]. The fact that graphene edges offer a quadrillion orders of magnitude larger specific capacitance in relation to double layer charge, the rate of faster electron transfer, including moderate electrocatalytic activity than the basal plane fully supports this conclusion [4]. Therefore, materials having some properties of ideal graphene such as advanced electrical and electronic conductivity resulting from large surface area, though with noticeable number of structural flaws are required for the majority of electrochemical devices [5]. Selective oxidation is one method that can be used to incorporate defects and favorable chemical moieties into graphene [6]. Manufacture of the reduced graphene oxide from graphene oxide, which is generated through chemical exfoliation of the oxidized form of graphite by electrochemical, chemical, or thermal methods is a more practical technique [7]. GO reduction, regardless of the specific reduction method, results in improved rGO capacitance and charge transfer capabilities as well as a lowered O/C ratio linked to improved conductivity [8]. To achieve ideal capacitance, one must, however, strike a compromise between the conductivity of reduced graphene oxide and quantity or amount of the oxygen functional groups [9]. Utilization of graphene-based composites in various electrochemical routines requires exact control and tuning of the chemical and structural properties of the Graphene Oxide [10]. However, this research demonstrated that they are easily accomplished via chemical reduction, which uses proper reduction procedures to regulate the final rGO degree of reduction [11].

Graphene Oxide (GO) can either be coated as a thin layer directly on the electrode surface or suspended in an electrolyte solution for electrochemical reduction [12]. While characterization is normally carried out using ex-situ techniques, the reduction has previously been conducted in both aqueous and non-aqueous electrolytes [13]. Different oxygen functional groups were demonstrated to be reduced at various potentials, and the influence of pH and solvent on the characteristics of the resulting final rGO was demonstrated [14]. The type of supporting electrolytes affects the complexity of the process. However, multiple approaches that offer a macroscopic average feature of the final rGO are commonly used to describe rGO films that are fabricated in different forms of reduction techniques [15].

In this article, we discussed the synthesis method for graphene oxide adopting improved Hummer's technique, then chemical reduction of the graphene oxide to achieve reduced graphene oxide as a final product.

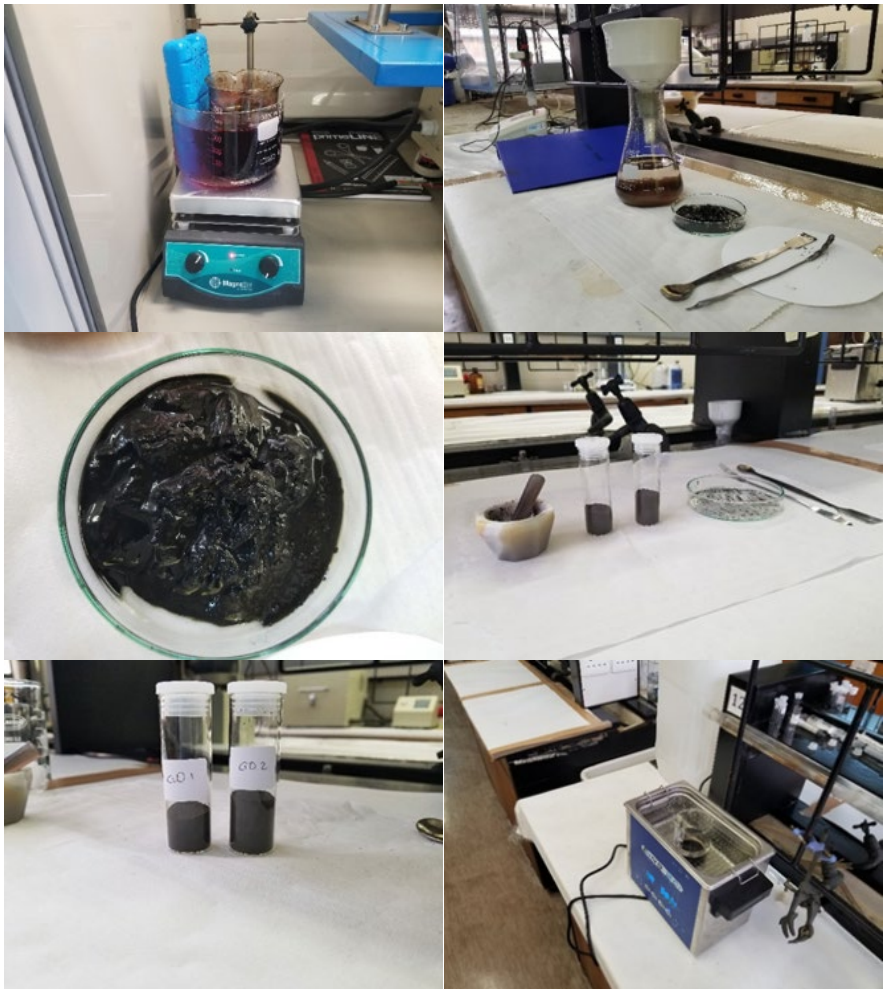

Figure 1. Synthesis of (a) GO, (b) filtration of synthesized GO, (c) green GO, (d) trituration using mortar and pestle, (e) the obtained GO samples and (f) chemical reduction of GO to rGO.

The average crystallite sizes were determined using the Scherrer equation as:

$$D = \frac{k\lambda}{\beta \operatorname{Cos}\theta}$$

Where D is the crystallite size, k Scherrer constant, λ the X-ray wavelength and β is the line broadening at FWHM.

The d-spacing values were also calculated with the help of Bragg's relation,

$$n\lambda = 2d\sin\theta$$

Where, n is the diffraction order, λ wavelength, d the interplanar spacing and θ is the diffraction angle.

2. Experimental

2.1. Chemicals, Reagents and Materials

Boric acid (H_3BO_3 99.5%) and Potassium permanganate ($KMnO_4$ 99%) were both

supplied from Central Drug House Pvt. Ltd. (CDH). Hydrogen peroxide (30%), hydrochloric acid (35%), and sulphuric acid (98%), were supplied by Merck. Fine powder of the Graphite 325 mesh (99.8%) was obtained from Alfa Aesar. 220 nm pore size vacuum filtration membrane with 47 mm diameter was obtained from Pall Corporation. Sodium hydroxide (NaOH) pellets were supplied by Fisher Scientific.

2.2. Equipment

The measurements for electrochemical processes were done with the help of Metrohm Autolab specifically PGSTAT 128N, constituting conventional three-electrode scheme. Modified glassy carbon electrode, Platinum wire, and Ag/AgCl/KCl (3.0 M) were employed for working, counter and reference electrodes correspondingly. Infrared spectra in addition to X-ray diffraction characteristics for both Graphene Oxide (GO) as well as reduced Graphene Oxide (rGO) were obtained with the help of Fourier transform infrared or FTIR spectrometer with model PerkinElmer spectrum 65 and the X-ray diffraction or XRD instrument with Cu-K (α) radiation at wavelength, $\lambda = 0.154$ nm with its model Miniflex 600. For uniform mixture formation of the dispersion a Sonicator with model Branson 2510 was employed. Vacuum oven with Model number OV-11/12 employed for boiling as well as drying purposes.

2.3. Graphene Oxide Synthesis

Graphene Oxide (GO), as a starting material for rGO fabrication was primarily produced using modified Hummer's process demonstrated elsewhere [16]. Pristine graphite powder, which weighed 1 g, was added to 25 ml H_2SO_4 under continuous stirring at 300 rpm for 2 h in 250 ml volume beaker. The reacting mixture temperature was held lower around 5˚C - 10˚C with the help of ice bath. 3 g of Potassium permanganate as an oxidizing agent was then gently added under uniform stirring of 300 rpm while the temperature of the system was monitored to be less than 10˚C. Following the incorporation of the potassium permanganate, the sample mixture was constantly stirred at the same speed of 300 rpm for 8 hrs. To avoid overheating water (125 ml) was then added to the batch size of 25 ml. Sonication and stirring of the mixture was done for 20 min period for each respective process and this sequence was done repeatedly 10 times for further exfoliation. Next, 15 ml of hydrogen peroxide was poured into the formed chocolate brown blend, covering the 250 ml beaker with aluminium foil to prevent photochemical degradation of the H_2O_2. At this step, the mixture becomes yellowish brown in colour with evolution of active bubbles. Following this step the obtained Graphene Oxide (GO) precipitate was cleaned primarily triple times using HCl having a concentration of 2 M then followed by triple times using ethanol, and several times using Deionized (DI) H_2O until the pH of resulting supernatant becomes 7 or neutral by means of centrifuging machine rotating at 8500 rpm for 45 min. Finally, the yield was allowed to dry at 60˚C in the vacuum oven.

2.4. Reduced Graphene Oxide Synthesis

Primarily, 0.75 g of sample Graphene Oxide (GO) powder was dispersed in 50 ml of DI H_2O followed by sonication for 2 hrs. Then KOH solution with 4 M concentration was prepared in 50 ml volume. The dispersion of GO prepared was added to this KOH and stirred for 1 h at 400 rpm. This composite blend was then moved to a stainless-steel autoclave (Teflon-lined) and placed into a heating oven at a temperature of 150˚C for eight hours. The obtained dark and black rGO material from these synthesis steps was finally collected using vacuum filtration procedure. Final washing process for rGO was performed by means of DI H_2O repetitively until the pH reaches 7 and again three times by using ethanol employing vacuum filtration for separation. The material was then allowed to dry in a vacuum oven at 120˚C until it was totally dried out.

3. Results and Discussion

3.1. XRD and FTIR Characterizations

The X-ray diffraction plots of rGO and GO are shown in Figure 1 below for comparison. The graphite is already known to have peaks with high intensity around 2θ of 24˚ and 26˚ then peaks with lower intensity at 2θ = 54.7˚. Following the oxidation process, however, *i.e.*, the oxidation of pristine graphite chemically to graphene oxide by following modified Hummer's procedure, the shifted X-ray diffraction peaks to the lower 2θ values, confirmed the internal structure of the graphite is altered because of the incorporation of new oxygen functional groups in addition to water molecules. The obtained Graphene Oxide (GO) exhibited sharp peak at 2θ = 11.38˚ and smaller peak at 2θ of 45˚ besides 80˚ respectively.

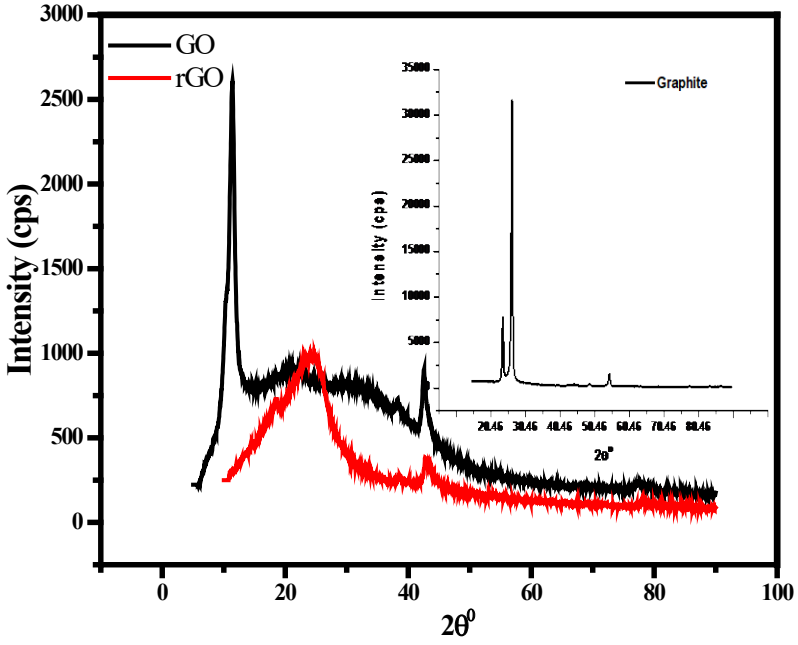

Figure 2. X-ray diffraction patterns of reduced Graphene Oxide (rGO) and Graphene Oxide (GO).

Furthermore, the reduced Graphene Oxide (rGO) confirmed that the observed peaks moved or shifted to the higher 2θ values when contrasted to those of Graphene Oxide (GO). This reveals that internal makeup of graphene is repositioned after partial reduction of certain oxygen functional groups in the internal structure. The reduced Graphene Oxide (rGO) exhibited a broad peak centered at 2θ value of 24.69˚ in addition to smaller peaks at 2θ values of 45˚ and 80˚ respectively. (See Table 1)

In addition to the X-ray diffraction results, the FTIR results demonstrated in Figure 2 further witnessed the chemically processed modification of graphite to Graphene Oxide (GO) and finally to reduced Graphene Oxide (rGO). Through the process which is known as chemical oxidation of graphite to graphene oxide, the peaks representing C=O appear near 1724 nm while –OH functional groups start to appear around 1411 nm and at 1219 nm correspondingly. Upon further reduction to reduced graphene oxide these indicated peaks disappeared.

Table 1. Average crystallite sizes computed for Graphite, GO and rGO.

	Graphite	GO	rGO
Average Crystallite size (D) in nm	32.90	8.15	1.19
d-spacing in nm	0.34	0.69	0.80

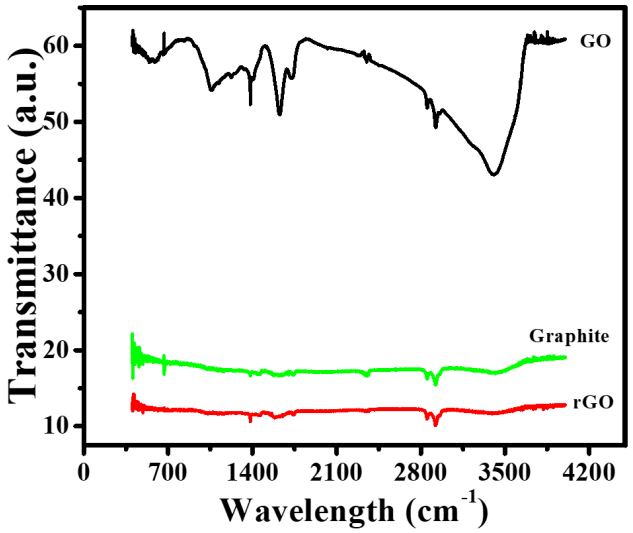

Figure 3. The plot of FTIR spectra for graphite, graphene oxide and reduced graphene oxide.

3.2. Electrochemical Characteristics of rGO, GO and Graphite with Modified Glassy Carbon Electrodes

The activities of the produced materials of graphene oxide, as well as reduced graphene oxide including the pristine graphite were examined by running the cyclic voltammograms in 0.5 mM of $K_3Fe(CN)_6$ mixed in 1.0 M KCl supporting electrolytic solution via modified glassy carbon electrode. Because of its sensi-

tivity to surface chemistry and microstructure the $K_3Fe(CN)_6$ redox couple was used. As is demonstrated in Figure 3, the oxidation-reduction peak currents for $K_3Fe(CN)_6$ were considerably higher for reduced Graphene Oxide (rGO) followed by graphite modified or casted Glassy Carbon (GC) electrode than Glassy Carbon (GC) electrode without modification commonly known as bare GCE. These peaks were revealed with the smallest value on Graphene Oxide (GO) modified Glassy Carbon Electrode (GCE) which resulted from the least electronic conduction characteristics of the Graphene Oxide (GO). Therefore, the electrochemical investigations further confirmed the internal structure transformations during the synthesis of graphene oxide and reduced graphene oxide from pristine graphite, and this is consistent with the spectroscopic results. (See Figure 4)

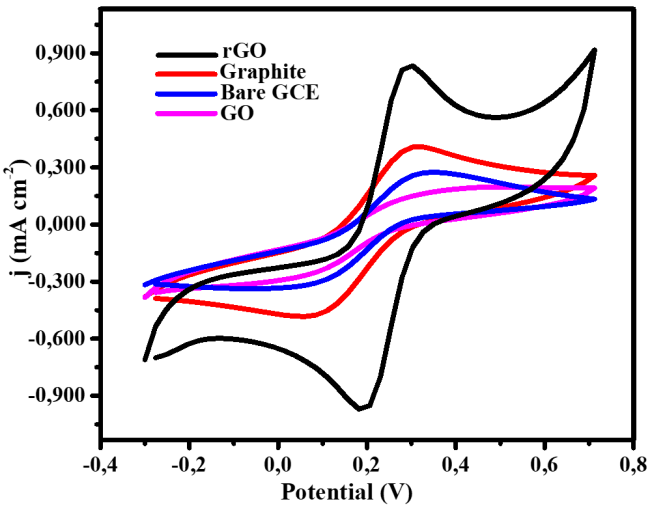

Figure 4. The cyclic voltammetry measures recorded on bare GC, GO, graphite, and rGO modified glassy carbon electrode in a 5.0 mM $K_3Fe(CN)_6$ + 1.0 M KCl electrolyte solution.

4. Conclusion

Graphene Oxide (GO) was synthesized through oxidizing process of pristine graphite via modified Hummer's approach. The findings from FTIR characterization revealed that the oxidation of the graphite has resulted from strong oxidants and the atoms of oxygen are placed into the graphite planes developing C=O and -OH bonds between graphene layers. Electrochemical behavior of Graphene Oxide (GO) modified Glassy Carbon Electrode (GCE) was studied with reference to K_3FeCN_6 redox electrochemical system reflects that the electrochemical characteristic is monitored by the electron transfer. Finally, an efficient, simple, and economical electrochemical method has been used to produce rGO. To end with, excellent electrochemical performance of rGO is observed via electrochemical method, so that rGO may be used as a promising electrode material for different practical applications, such as metal-air batteries, supercapacitors and energy conversion devices.

Conflicts of Interest

The authors declare no conflicts of interest.

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