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Review of Fascial Synthesis Route of Graphene Oxide by Using Equivalent Hummer Method

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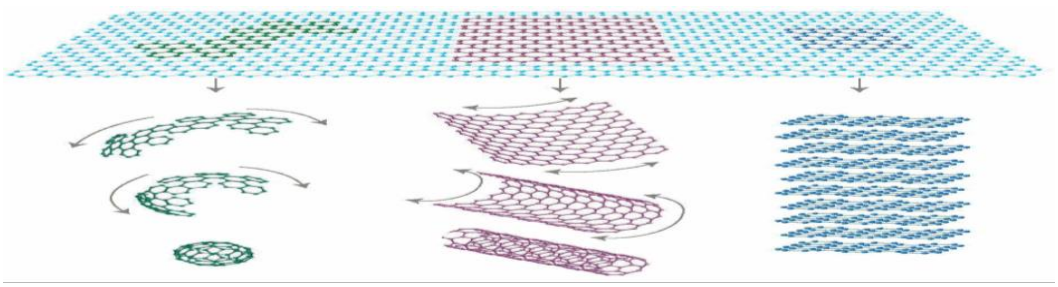
ABSTRACT

Graphene oxide is now a valuable substance in research and technology. We are concentrating on graphene oxide, GO has unique mechanical, optical, and electrical properties, making it a promising material for a wide range of applications, including energy storage, catalysis, sensing, and biomedical devices. We have studied here highly oxidised, less hazardous, temperature-controlled graphene oxide compounds. These significant materials are synthesized by using a variety of methods, such as the Hummers Method, the Modified Hummers Method and the Improved Hummers Method. We have implemented here equivalent Hummer's methods for graphene oxide, this material has maximum utility in advanced nanomaterial science. It is also highly tuneable, with its properties depending on the degree of oxidation and the type and number of functional groups present on its surface. Due to its ease of production and modification, GO has become a popular research topic in recent years, and its potential for various applications is being explored extensively.

Introduction:

Carbon structure study is much sizeable due to its unequalled properties and numerous applications. Along different category of carbon, graphene is mostly noted due to its remarkable excellent properties [1,2] Graphene is a stimulating material. Graphite having single atomic layer is known as graphene. Thickness of graphene is of single carbon atom arranged in honeycomb lattice. They are much solid and can be designed into zero dimension, 1-d,2-d,3-d forms[3]. Graphene has the two dimensional honeycomb lattice accelerate sp^2 hybridization which, leads to remarkable mobility near 100,000 cm^2/Vs at room temperature [4]. Graphene has the young modulus of 1TPa and tensile strength 130GPa [5] It also have unusual thermal conductivity 5300 W/ mK [6], due to which it has great potential in electronic sector such as transistor and integrated circuit [7], energy storage [8] gas sensor [9] bio electronic sensor [10]. Graphene is transferable two dimensional single layer nanosheet was first obtained by mechanical exfoliation (Scotch-tape method) of bulk graphite [11] and it was also done by epitaxial chemical vapour deposition [12]. Although those routes might be propose for specific device assembly, they can be less fruitful for large scale manufacturing. Chemical means are a practical approach to synthesis bulk scale graphene material [13]. In synthesis of graphene scalability is an important factor and one of

the most popular process for graphite exfoliation is use of strong oxidizing agent to obtain Graphene oxide (GO) , to obtain nonconductive hydrophilic carbon material. [14 15]. Although it is difficult to determine the exact nature of graphene oxide (GO), it is clear that GO the previously contagious aromatic lattice of graphene interrupted by epoxies, alcohols, ketones, and carboxylic groups. [16-18]. The disorder of lattice give back an increase in interlayer spacing from 0.335nm for graphite to more than 0.625nm for GO [19] the powder of natural flake graphite (NFG) can easily be oxidized to produced GO. Due to its inexpensive cos, vast availability, and ease to convert to graphene, GO is of great interest.



0-D Bucky Ball **1-D Carbon Nanotube** **3-D Graphite**

Fig.1 Various structure of graphene (0-D Bucky Ball, 1-D Carbon Nanotubes, 3-D Graphite) Reproduced with permission from ref **20** (Copyright 2016 springer publications)

Experimental Details

Hummers' Method

Involved adding concentrated H_2SO_4 (69 mL) to a mixture of graphite flakes (3.0 g, 1 wt equiv) and $NaNO_3$ (1.5 g, 0.5 wt equiv) and cooling the mixture to 0 °C. To keep the reaction temperature below 20 °C, KMnO4 (9.0 g, 3 wt equiv) was added slowly in parts. The reaction was warmed to 35 °C and agitated for 30 minutes before progressively adding water (138 mL), resulting in a large exothermic to 98 °C. External heating was used to keep the reaction temperature at 98 °C for 15 minutes before it was removed and cooled in a water bath for 10 minutes. Another exothermic was produced after adding more water (420 mL) and 30% H_2O_2 (3 mL). After air cooling, the mixture was refined as described above for the IGO (sifting, filtration, numerous washings, centrifugation and decanting, vacuum drying) to yield 1.2 g of solid.

Improved Hummers' Methods

A 9:1 mixture of concentrated H_2SO_4/H_3PO_4 (360:40 mL) and a mixture of graphite flakes (3.0 g, 1 wt. equiv.) and $KMnO_4$ (18.0 g, 6 wt. equiv) were added for the improved procedure, which resulted in a minor exothermic to 35-40 °C. After that, the reaction was heated to 50 °C and stirred for 12 hours. The reaction was brought to room temperature and poured onto 400 mL of ice with 30% H_2O_2 (3 mL). For workup, the mixture was filtered through polyester fibre after being sifted through a metal U.S. Standard testing sieve (W.S. Tyler, 300 m) (Carpenter Co.) After centrifuging the filtrate at 4000 rpm for 4 hours, the supernatant was decanted out and discarded. The remaining solid matter was next washed sequentially with 200 mL of water, 200 mL of 30% HCl, and 200 mL of ethanol (2) for each wash, the mixture was then sifted through a U.S. Standard testing sieve and filtered through polyester fibre, with the filtrate being centrifuged (4000 rpm for 4 h) and the supernatant decanted away. After this prolonged, multiple-wash operation, the material that was left over was coagulated with 200 mL of ether, and the resulting suspension was filtered onto a PTFE membrane with a 0.45

m pore size. 5.8 g of product were produced after the solid on the filter was vacuum-dried for a whole night at room temperature.

Modified Hummers Method:

This process is referred to as Modified Hummers' method, and the end result as HGO. Graphite flakes (3.0 g, 1 wt. equiv.), $NaNO_3$ (1.5 g, 0.5 wt. equiv.), and concentrated H_2SO_4 (69 mL) were combined, and the mixture was chilled in an ice bath to. 0 °C. To keep the reaction temperature below 20 °C, $KMnO_4$ (9.0 g, 3 wt. equiv.) was added gradually. For 7 hours, the reaction was stirred and warmed to 35 °C. The reaction was agitated for 12 hours at 35 °C with the addition of further $KMnO_4$ (9.0 g, 3 wt. equiv.). Using 400 mL of ice and 30% (3 mL) H_2O_2, the reaction liquid was emptied after being cooled to room temperature Following the preceding procedure of sifting, filtration, centrifugation, decanting with numerous washes, and final vacuum drying to produce 4.2 g of solid product, the mixture was next refined.

Equivalent Hummers Method:

Graphite powder and sodium nitrate were used in a ratio of 1.2 to synthesise graphene oxide. Using a conical flask and 75ml H_2SO_4, the setup was cooled using an ice bath. Slowly adding $KMnO_4$ 6 gm while keeping the temperature below 20 °C 3 hours were spent stirring the reaction at less than 20 °C. In it, the viscus slurry developed. The ice bath was removed, 70 ml of D.W. was added, and the temperature was kept below 10 °C. In order to eliminate the metallic ions from the process, 3 ml of H_2O_2 was supplied after adding 150 ml of D.W steadily over the course of 15 minutes at 60 °C. As graphene oxide developed, it turned from dark brown to golden yellow in colour. Moreover, the stirring was continued for an additional 2 hours and 20 degrees Celsius. After being centrifuged at 5000 rpm for 20 minutes, the solid was cleaned with 10% HCl, centrifuged once more at 10,000 rpm for 15 minutes, washed with DW water, and dried in an oven at 60 °C for 12 hours. It creates the powdered graphene oxide.

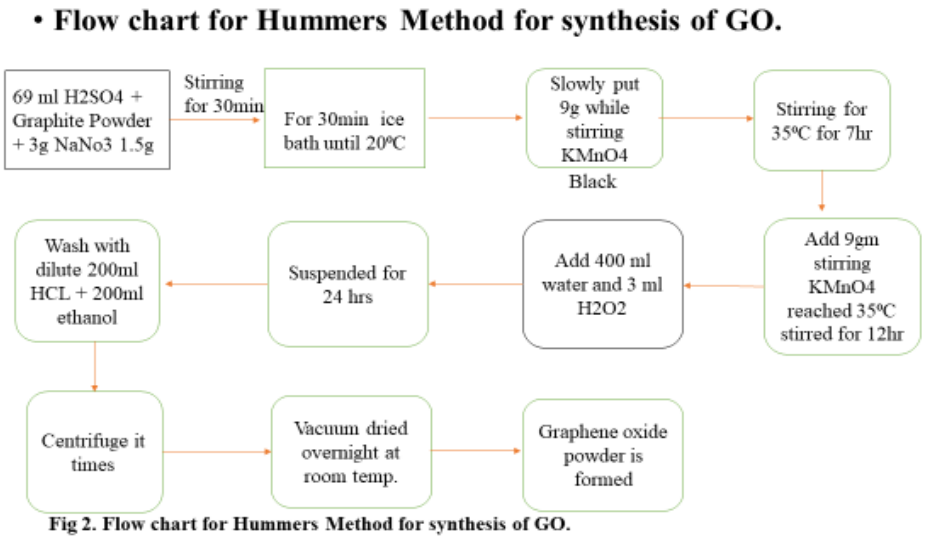


Fig 2. Flow chart for Hummers Method for synthesis of GO.

• **Flow chart for Improved Hummers Method for synthesis of GO.**

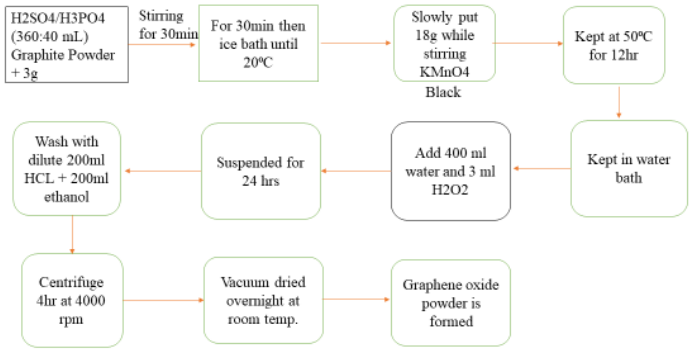


Fig 3. Flow chart for Improved Hummers Method for synthesis of GO.

• **Flow chart for Modified Hummers Method for synthesis of GO.**

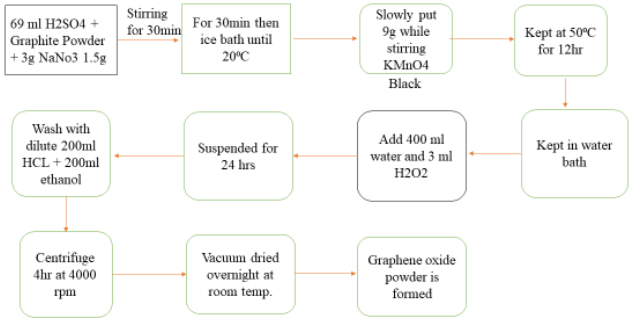


Fig 4. Flow chart for Modified Hummers Method for synthesis of GO.

• **Flow chart for Equivalent Hummers Method for synthesis of GO.**

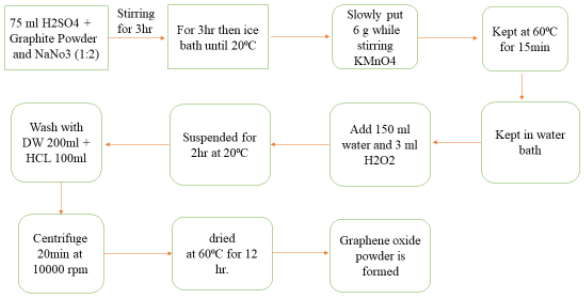


Fig 5. Flow chart for Equivalent Hummers Method for synthesis of GO.

Fig.6: GO appears in Green Colour through Equivalent Hummer Methods

Structure of Graphene Oxide:

Graphene oxide was fabricated much after than graphene, a material that has generated such great scientific interest. Graphene oxide is a monolayer of carbon atoms having both (significantly) sp2 hybridized carbon atoms and (partially) sp3 hybridized carbon atom bearing oxygen containing functional groups located both on the basal (hydroxyl and epoxy) and edge plane carboxyl, carbonyl). Carbon atoms covalently bonded to oxygen containing functional groups are sp3 hybridized carbon cluster are located slightly above and below the plane of sp2 hybridized carbon atom which is conformed from HRTEM. [21 22]. We are going to confirm this atomic layer at angstrom AFM atomic force microscopy and this AFM atomic micro graph confirms the formation of graphene oxide structure. Small amount of oxygen mass reduced is very dilute chemicals reaction at room temperature.

This LK model has become one of the most widely accepted and used for moderately oxidised GO.

Fig,7: Lerf- Klinowski model of graphene oxide

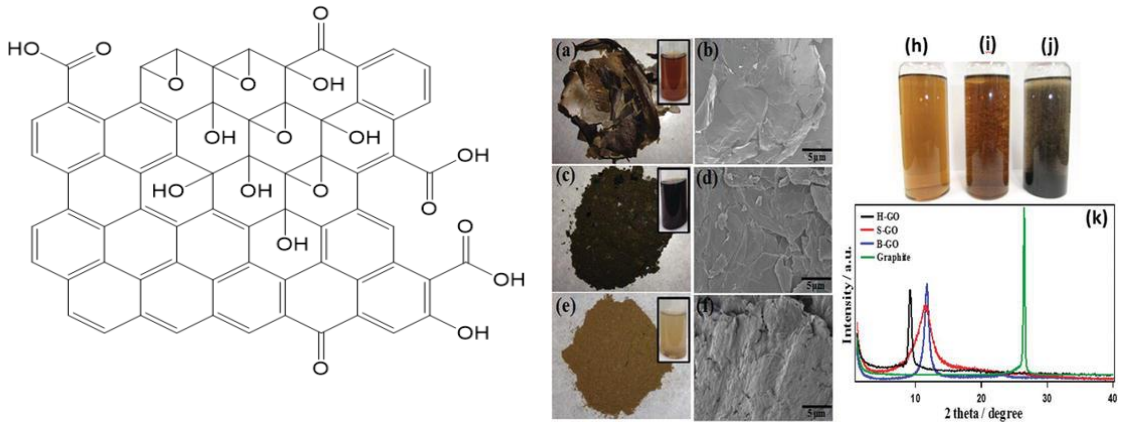


Fig.8: GO powder and nano dispersed in chemical form with surface morphology of SEM micrograph with verified XDR results adapted from this reference, https://www.intechopen.com/chapters/54591.

Conclusion:

Graphene oxide (GO) was successfully prepared by equivalent hummer method. Fascial synthesis route of GO can be the most suitable and prominent material than hummer modified, improved hummers and modified hummers method. Due to its ease of production and modification of equivalent hummers method, GO has become appear in green colour powder and nano dispersed in chemical form that compare with consonant fairly reported results GO powder and nano dispersed in chemical form with surface morphology of SEM micrograph with verified XDR results adapted from this reference, https://www.intechopen.com/chapters/54591.

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