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PREPARATION OF GRAPHENE OXIDEFROM GRAPHITE POWDER USING HUMMER'S MODIFIED METHOD

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Abstract: The modified Hummer process was used to successfully make graphene oxide (GO) films witha two-dimensional structure. The redox approach has been shown to be a viable method for large-scale GO film production. The characteristics of GO films have been thoroughly characterized. The existence of oxygen-containing functional groups in GO films was confirmed by a Fourier-transform infrared spectrum analyzer (FT-IR). The UV-VIS spectrometer revealed that the GO sheet had excellent optical responsiveness. In this study, thechemical structure of GO sheet was also reported. There is a discussion and references for moregraphene research.

Key words: Graphene Oxide, Hummer, Redox approach, GO films.

1. INRODUCTION

Graphene is a material made up of carbon atoms linked together in a repeating hexagonal pattern. Because graphene is extremely thin, it is classified as a two-dimensional material. Graphene is considered the strongest material in the world, as well as one of the best conductorsof electricity and heat. Graphene has a multitude of potential applications, in almost every industry (such as electronics, medicine, aeronautics, etc.).

Since graphene is expensive and relatively difficult to produce, great efforts are being made tofind efficient but inexpensive ways to produce and use derivatives of graphene or related materials. One such material is graphene oxide (GO). It is a monoatomic layered material, produced by strong oxidation of graphite, cheap and abundant. Graphene oxide is an oxidizedform of graphene, interwoven with oxygen-containing groups. Because it disperses in water (and other solvents), it is considered simple to treat, and it can even be used to make graphene.Graphene oxide is not a good conductor of electricity, but processes exist to increase its properties. It is commonly sold as a powder, dispersed or as a garnish.

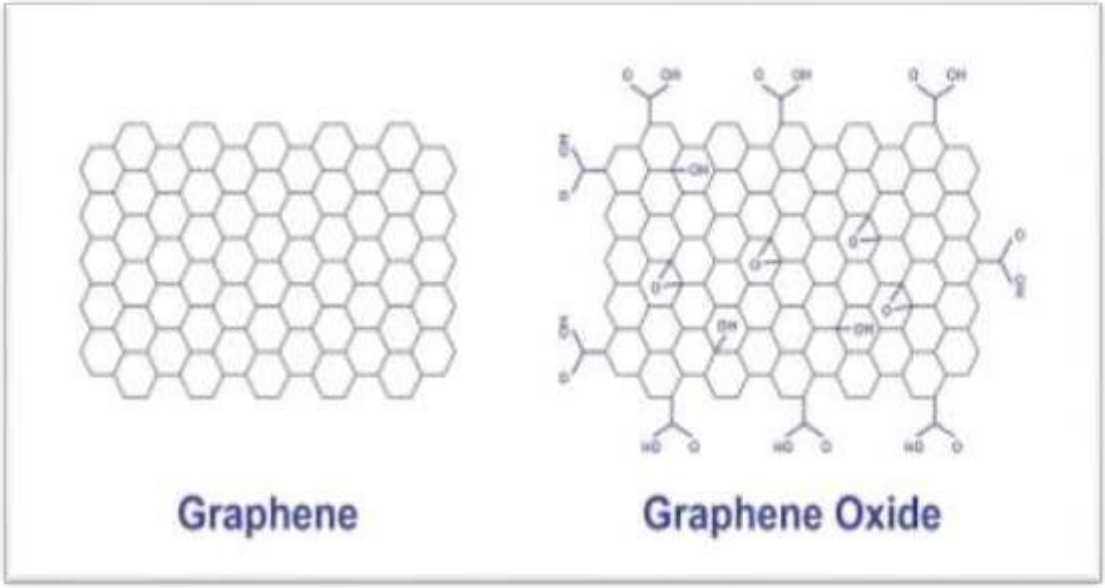

Fig 1.1 Graphene & Graphene oxide structure

Graphene oxide is synthesized by four basic methods: Staudenmaier, Hofmann, Brodie and Hummers. Many variations of these methods exist, with continual improvements being discovered for better results and less expensive processes. The carbon / oxygen ratio of graphene oxide is commonly used to assess oxidation efficiency.

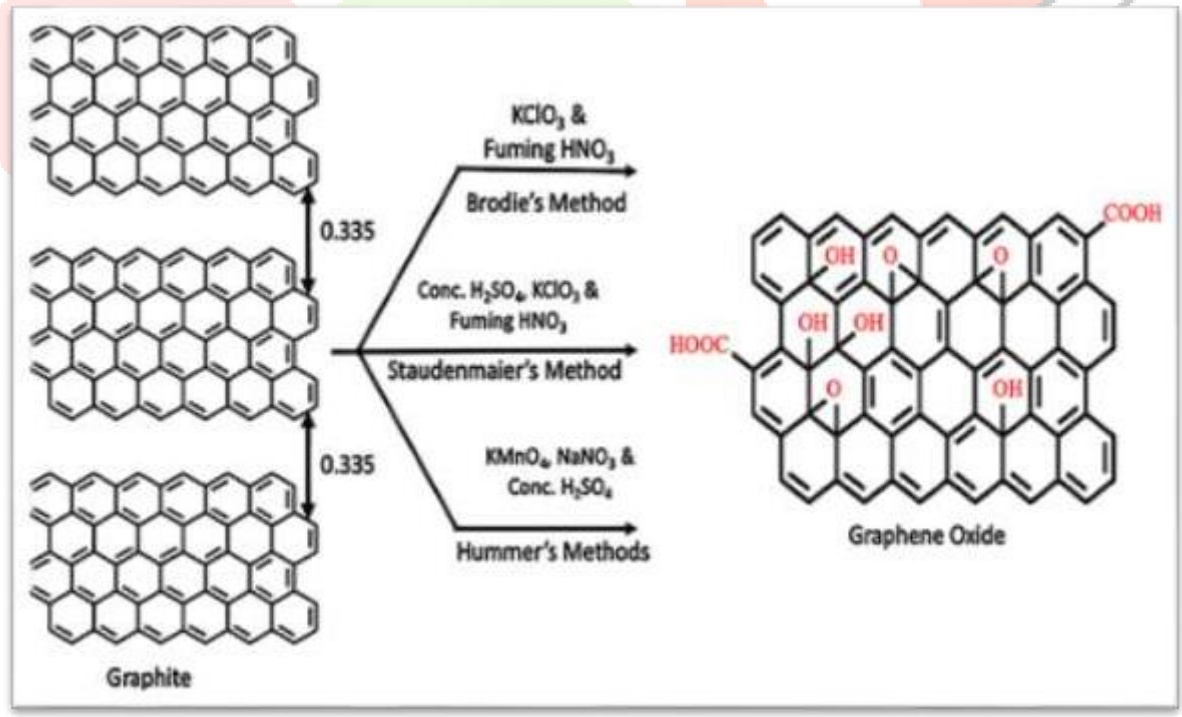

Fig 1.2 Different methods for graphene oxide preparation.

GRAPHENE OXIDE (GO) is graphene in its oxidized state. It is a single-atomic-layered substance created by the oxidation of cheap and widely accessible graphite. Graphene oxide issimple to work with because it dissolves in water and other solvents. Graphene oxide is not conductive due to the oxygen in its lattice, but it can be converted to graphene via chemical techniques.

Solution-based approaches can be used to process graphene oxide.
One of the most significant advantages of graphene oxide is that it is water dispersible. This enables the use of solution-based methods. Chemical vapor deposition is the most common way to make graphene films (CVD). However, because this process necessitates high temperatures and extensive deposition times, it is costly. It also restricts deposition to substrates that can withstand high temperatures, making polymer deposition challenging.

Spray, spin, and dip coating, as well as Langmuir-Blodgett (LB) depositions, are all solution- based techniques. It is possible to convert graphene oxide to graphene.

Another advantage of graphene oxide is that it can be converted to graphene via chemical, thermal, or electrochemical means. Reduced graphene oxide is the name given to the substancecreated (rGO). When vast amounts of graphene are required for industrial applications like as energy storage, the rGO is one of the most obvious choices. As a result, the reduction processis critical since it has a significant impact on the quality of the rGO generated.

Chemical reduction is a fairly scalable method of reducing GO, however the quality of the rGOgenerated is often low. Thermally decreasing GO necessitates temperatures of 1000 °C or higher, which degrades the structure.

Once reduced graphene oxide has been created, it can be functionalized in a variety of ways. This will improve the rGO film's characteristics, allowing it to be employed in a variety of applications.

1.1. Properties of graphene oxide

Graphene is a bicyclic crystalline allotrope with a hexagonal lattice structure made up of purecarbon atoms.

They are best known for their unique properties of high optical transparency, better thermal conductivity at room temperature and flexibility in all resistant nanoscale materials. Mechanically exfoliating 3D graphite crystals and peeling off a single sheet of graphene with tape was the initial method of discovering graphene. Since then, graphene's qualities have been

identified, and several ways have been tried to determine the best approach to generate huge amounts at low cost, although there are still many problems to overcome with these specs.

Graphene oxide (GO) is an excellent precursor for obtaining graphene with higher yield and lower cost. Graphite oxide is made from graphite crystals that have been oxidized with powerful oxidizing agents like sulfuric acid. Using ultrasound, graphite adopts oxygen-retaining functional groups allowing the material to disperse in water while increasing the distance between layers.1 Graphite oxide can then be exfoliated to form oxide mono or multilayer graphene (GO). The difference between graphite oxide and GO is based on their different structures but the chemical composition remains the same. GO is a monomeric material made up of molecules of carbon, hydrogen, and oxygen that eventually becomes cheapbut still plentiful. To counter this discontinuity, GO can be reduced to form reduced grapheneoxide (rGO) to regain One of the most important features of GO is that it can be produced usinggraphite (because it's cheap) using different chemical methods, resulting in high yields with farsuperior cost effectiveness.

Although the surface of these GO sheets has some flaws, the overall unit cell size is still very similar to that of graphene.2 GO is therefore an oxidized version of graphene consisting of oxygen-containing groups. . Due to the presence of different functional groups, GO has a lowerelasticity and its Young's modulus depends on the functionalization and molecular structure ofthe functional

Graphene oxide may easily disperse in organic solvents, water, and other matrixes because to the presence of oxygen functions. This is a major benefit when combining the material with polymer or ceramic matrixes to enhance their mechanical and electrical properties.

Because of the disruption of its sp2 bonding networks, graphene oxide acts as an electrical insulator in terms of electrical conductivity. In order to restore electrical conductivity, it is necessary to reduce graphene oxide and regain the honeycomb hexagonal lattice of graphene.

It's difficult to scatter reduced graphene oxide (rGO) once a substantial number of oxygen groups have been removed since this material prefers to aggregate.

Table 1.1 Properties of Graphene, Graphene oxide, reduced graphene oxide

Properties	Graphene	GO	rGO
Electron mobility @ room temperature	~200,000–250,000 $cm^2 V^{-1} s^{-1}$	0.1–10 $cm^2 V^{-1} s^{-1}$	2–200 $cm^2 V^{-1} s^{-1}$
Surface area	2630 $m^2 g^{-1}$ [38]	736.6 $m^2 g^{-1}$ [52]	466–758 $m^2 g^{-1}$ [53,54]
Thermal conductivity	~5000 $W m^{-1} K^{-1}$ [38,54]	0.5–18 $W m^{-1} K^{-1}$ [55,56]	1390–2275 $W m^{-1} K^{-1}$ [56–58]
Carbon-carbon bond length	0.142 nm [54–59]	N/A	N/A
Specific capacitance (depends on cyclic voltammetry)	550 $F g^{-1}$ [60]	215–255 $F g^{-1}$ [61]	210–425 $F g^{-1}$ [60,62,63]
Electrical conductivity (depends on reduction technique)	~6×10^8 $S m^{-1}$ [53,64]	5.7×10^{-6} $S m^{-1}$ [62]	10^2–10^5 $S m^{-1}$ [65–67]
Sheet resistance	200 Ωsq^{-1} [68]	~10^{10}–10^{12} Ωsq^{-1} [51]	~10^2–10^6 Ωs^{-1} [51,67]

The functionalization of graphene oxide can alter the characteristics of graphene. The chemically-altered graphene's obtained by this method could possibly be used in several applications. Graphene oxide can be functionalized in a variety of ways, depending on the application.

Using amines through organic covalent functionalization is one technique to ensure that chemically changed graphene scatter freely in organic liquids, for instance. This makes the material better suited to production of bio devices and optoelectronics, and for use in drug delivery

It has also been demonstrated that fullerene-functionalized secondary amines and porphyrin- functionalized primary amines can be attached to graphene oxide platelets to improve the material's nonlinear optical performance.

In the fabrication of single layer or few-layer graphene sheets, graphene oxide could be employed as an intermediate. To accomplish this, an oxidation and reduction technique that can isolate carbon layers and separate them without affecting their structure should be devised.

Chemical reduction of graphene oxide is rated one of the most viable approaches for mass synthesis of graphene. However, scientists have struggled to mass-produce graphene sheets ofthe same quality as those created by mechanical exfoliation on a large scale.

1.2. Graphene oxide applications.

Graphene Oxide sheets can be deposited on almost any substrate and transformed into a conductor subsequently. As a result, GO is particularly well suited to the fabrication of transparent conductive films, such as those used in flexible electronics, solar cells, chemical sensors, and other applications. GO is even being researched as a replacement for tin oxide (ITO) in batteries and touch panels. Graphene oxide can be utilized as an electrode in batteries,capacitors, and solar cells due to its enormous surface area. Graphene Oxide is less expensiveand easier to produce than graphene, therefore it might be mass produced and used sooner.

GO can be easily blended with a variety of polymers and other materials to improve compositematerial qualities such as tensile strength, elasticity, conductivity, and more. Graphene Oxide flakes adhere to one another in solid form to form thin, stable flat structures that can be folded,wrinkled, and stretched. These graphene oxide structures can be employed for a variety of purposes, including hydrogen storage, Nano filtration membranes and ion conductors.

As graphene oxide is fluorescent, it's ideal for a variety of medicinal uses. Bio sensing and disease detection, medication transporters, and antimicrobial materials are just a few of the biological applications of GO.

Graphene oxide (GO), a kind of graphene with oxygen-containing groups, has been the subjectof a lot of discussion and conjecture recently, with the majority of it concentrated on its possiblemedical applications.

GO membranes are considered promising materials for water treatment applications due to their stability and high presence in water. As a result, tremendous progress has been achieved in producing high-performance membranes with a high rejection rate in recent years.

2. LITRETURE REVIEW

J.Song, X. Wang et.al "Preparation and Characterization of Graphene Oxide", journal of Nanomaterials. They prove that redox method is promising way to synthesize GO films on large scale they selected X-ray diffraction (XRD) to measure the crystal structure of GO sheet. Fourier-transform infrared spectra analyzer (FT-IR) to certify the presence of oxygen-containing functional groups in GO films. UV-VIS spectrometer and TGA analyzer that GO sheet possessed excellent optical response and outstanding thermal stability.

J.Chen, B.Yao et.al "An improved Hummers method for eco-friendly synthesis of graphene oxide" Journal of Carbon v.64, pp 225-229. They used improved Hummers method without using $NaNO_3$ to produce graphene oxide nearly same as that prepared by convectional Hummers method they proved this modification does not decrease the yield of product, eliminating the evolution of NO2/N2O4 toxic gasses and simplifying the disposal of waste water because of the inexistence of Na+ and NO3 ions. For the first time, they also developed a prototype method of post-treating the waste water collected from the systems of synthesizing and purifying graphene oxide.

W. S. Hummers Jr. and R. E. Offeman, "Preparation of graphitic oxide," Journal of the American Chemical Society, vol.80, no.6 , p.1339,1958. They developed a rapid relatively safe method for preparation of Graphitic oxide from graphite in what is essentially an anhydrous mixture of sulfuric acid , sodium nitrite and potassium permanganate

3. EXPERIMANTEL SECTION

3.1. Raw materials

Graphite powder purchased from Neelkanthhari Chemical, Vadodara, Gujarat. Sulfuric Acid (H_2SO_4), Sodium nitrite ($NaNO_3$), Potassium permanganate ($KMnO_4$), Hydrogen Peroxide (H_2O_2),

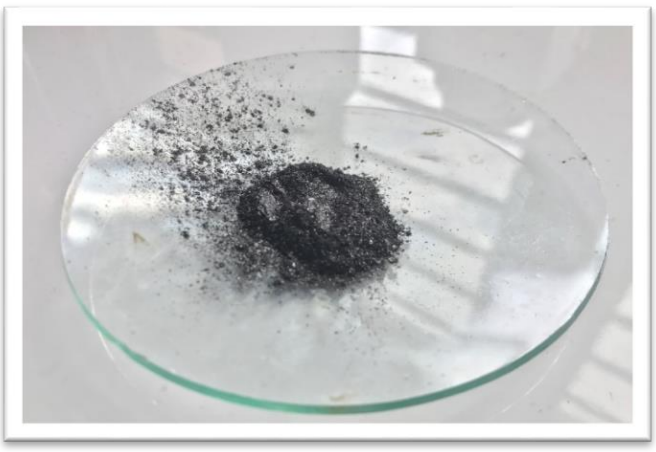

Fig 3.1 Graphite powder

3.2. Apparatus

Beaker, Water bath apparatus, Ice bath apparatus, Magnetic stirrer, Hot air oven, volumetric flask, Round Bottom flask, Measuring cylinder, Centrifuge.

3.3. Synthesis of Graphene Oxide: -

Graphene oxide was synthesized by using Hummer's modified method. 0.5g graphite powder and 0.5 g Sodium nitrite ($NaNO_3$) is added to 23.1Ml of 24N Sulfuric acid (H_2SO_4), in beaker after it is stirred in ice bath for 15minuts. Then 4.0g of Potassium permanganate ($KMnO_4$) was slowly added to in ice bath to yield purple green mixture. Then mixture was transferred to 40 C water bath and magnetically stirred for 90 minutes. The dark brown colored paste was diluted with the slow addition of 50 mL of deionized water (DI) and allowed to stir for a further 10 min.To make a golden-brown solution, a 6 mL amount of H2O2 was gently added to quench the solution. A further 50 mL of DI water was added, and the resultant product centrifuged and washed with warm DI water repeatedly to adjust the pH to ~6. The product was then dried for 24 hours at 80 degrees Celsius.

Fig 3.2 Graphene Oxide

4. Result and Discussion

The Optical Absorption Properties of GO Sheet. The analysis of UV-VIS diffuse reflectance spectra of the GO sheet was shown in Figure4.2. It is indicated that graphene oxide possessed a good absorption in the visible range (200~800 nm), but absorption in the ultraviolet range was also slightly decreased. The results showed the good photo response of GO sheet not only in ultraviolet range but also in visible range, which implied the enormous potential for application of light

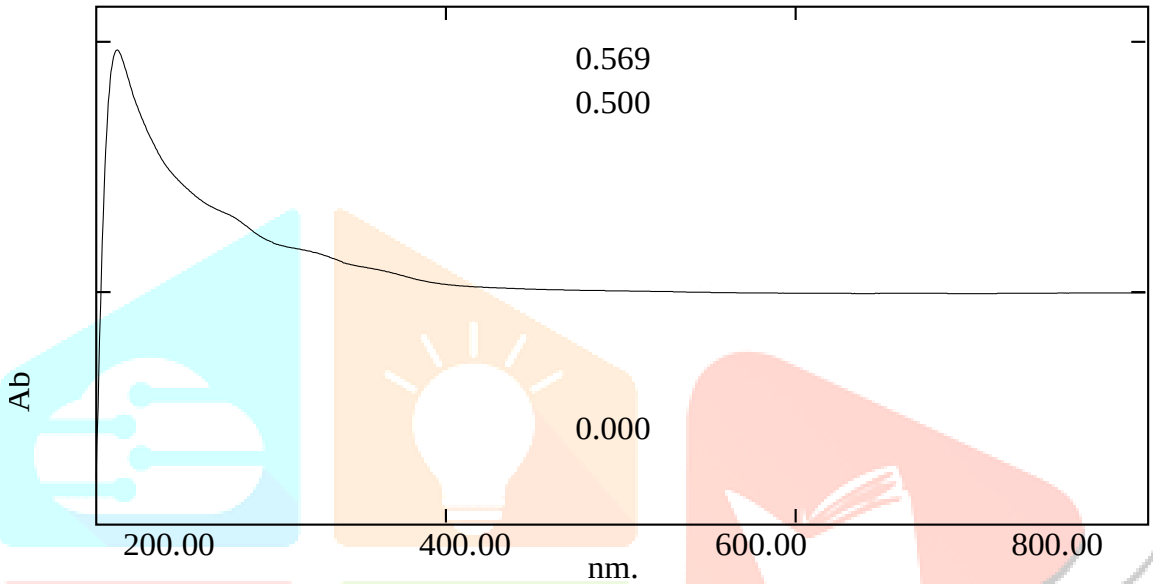

Fig 4.1 The UV-VIS spectra of GO.

No.	P/V	Wavelength	Abs.	Description
1	↑	212.00	0.483	

Table 4.1 UV-VIS spectra wavelength of GO.

4.2FTIR spectra analysis was performed to investigate the structure and functional groups of the materials. The GO showed apparent adsorption bands for the carboxyl C=O (1646 cm−1),

aromatic C=C (1509 cm−1), epoxy C–O (1488 cm−1), alkoxy C–O (1351 cm−1), and hydroxy –OH (3373 cm−1) groups. The presentation of oxygen-containing functional groups, such as C=O and C–O, further confirmed that the graphite indeed was oxidized into GO. The presentation of C=C groups showed that even graphite had been oxidized into GO; the main structure of layer graphite was still retained. The results FT-IR synthesis further demonstrated the successful synthesis of GO sheet.

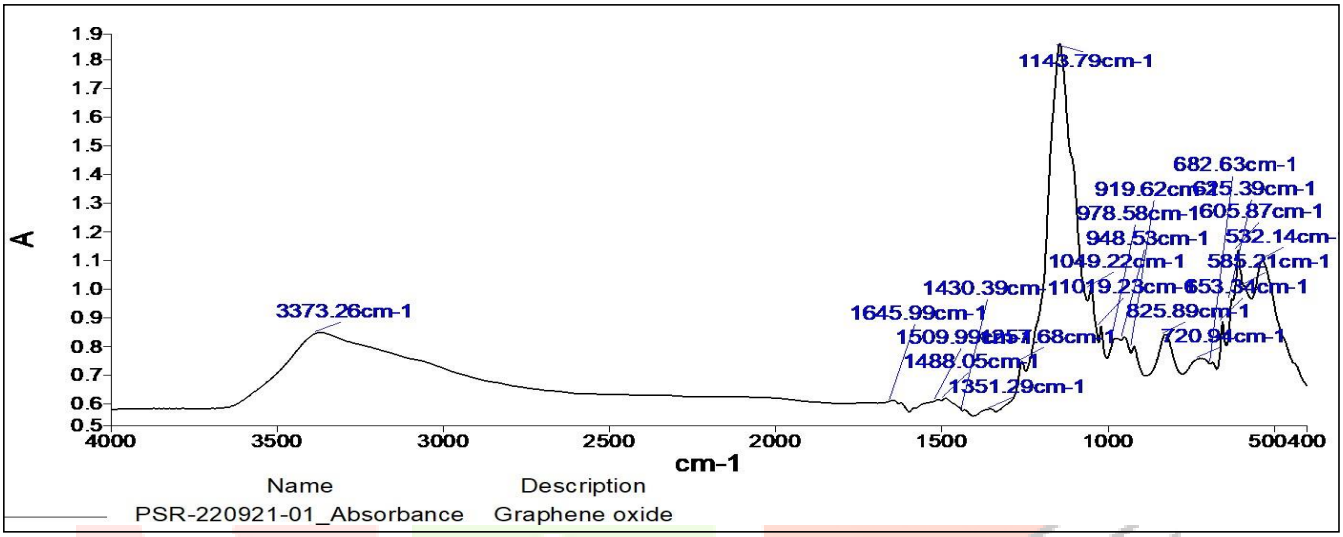

Fig 4.3 FT-IR Absorbance pattern of GO.

5. CONCLUSION

GO films were successfully prepared via modified Hummer method. The presence of oxygen-containing groups and characteristic peaks in FT-IR determined the successful preparation of GO sheets. The presence of oxygen-containing functional groups provided more opportunities for potential applications of GO in many areas. These data will provide a reference to further study the nature of graphene and graphene oxide.

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