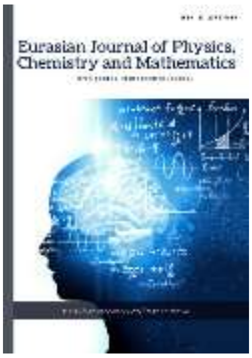

Synthesis and Characterization of Graphene Oxide. Review 2014-2020

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ABSTRACT

In recent years, graphene oxide has received much attention from researchers. Due to its multiple applications, including heavy metal adsorption, dye adsorption, microwave absorption, photocatalysis, sensing applications, and membrane separation processes, GO can be synthesized using a number of methods. According to Staudenmaier's oxidation methods, graphite oxide is formed by oxidizing graphite with concentrated acids or strong oxidants. The Hummer's method is widely used in the world because it has clearly explained the structure of graphite oxide using a variety of different models, as shown by Dreyer et al. The Lerf-Klinowski model, which is widely used in graphite oxide synthesis, has layers with hydroxyl and epoxy groups in the center and carboxyl groups at the edges. The oxygen groups in graphene oxide give it a hydrophilic character, and active groups contained between the layers give it an internal space of up to 6–12 A°, depending on the amount of water applied. This review highlights the study of the literature from 2014 to 2020 regarding the preparation and characterization of graphene oxide.

Keywords: Graphene Oxide, Hummer's method, electrical properties, reduced graphene oxide.

1. Introduction :-

Multiple studies around carbon led to a flourish in the field of nanotechnology. Diamond and graphite are three-dimensional types of carbon (known as allotropes). Carbon in its three-dimensional graphitic form has been documented since the 1500s [1,2]. When the pencil industry grew in the 1900s, this led to its use as a writing material [3]. Other carbon types as fullerene(azero- dimensions 0D), carbon nanotubes that are one dimensional (1D) [1-2]. In the 1980s and 1990s, they were discovered. However, there was a debate about the presence of a two-dimensional (2D) allotrope of carbon [4]. That was until 2004, when Andre Geim and Konstantin Novoselov published a paper that documented that case. They isolated a single layer of graphene on a sticky tape using micromechanical cleavage (scotch tape method) of highly ordered pyrolytic graphite [4]. A hexagonal honeycomb network of covalently connected sp2-hybridized carbon atoms makes up graphene, a single-layer, two-dimensional (2D) material. It's a one-atom thick carbon allotrope that forms the structural foundation for the rest of the carbon family: 1) Graphene sheets are wrapped into spheres to make 0D fullerene (bucky balls). 2) CNTs are made by rolling graphene sheets into cylindrical shapes, while 3D graphite is created by collecting multiple layers of separate graphene sheets joined by Van der Waals bonds [5]. Graphitic layers are typically found in single, double, and triple configurations. It's called graphene and is divided into three layers: Monolayer, bilayer, multilayer graphene or thick graphene is a term used to describe graphene with layers ranging from 5 to 30 [6], with a layer thickness of 0.33 nm, the carbon-carbon bond distance in graphene is around 0.142 nm. [3]. Theoretically, graphene has a large specific surface area (2630 m2/g) and other unique properties [7-9]. It has a high intrinsic mobility (200,000 cm2V-1s-1). Young's modulus is strong (1.0 TPa) , and (~5000 Wm−1K−1) thermal conductivity [10-11]. It also possesses a 97.7% optical transmittance, is electrically conductive, and can handle current densities of 108 A/cm2 [12], because graphene is a zero-band gap semiconductor, it can have its band gap changed using basic physicochemical approaches [13]. Study of graphene and its derivatives has gained a lot of interest in recent years due to its interesting properties, with a range of applications like membranes [14,15], nanoelectronics [16-18], Li-ion batteries [19], sensors, drug delivery, electrodes and super capacitors [20-25]. In comparison to graphene (G), graphene oxide (GO) offers the advantages of inexpensive production costs, large-scale production, and ease of processing. It's frequently utilized to initiate the manufacture of reduced graphene oxide (rGO) [26] . Scientists have discovered that GO possesses great features, including a significant number of active oxygen-containing functional groups, as a result of additional research in recent years [27]. Depending on the needs of specific application domains, these oxygen-containing groups or decreased doping components could be exploited as catalytic active centers for covalent/non-covalent modification design. Furthermore, the graphene oxide interlayer distance is widened when oxygen-containing groups are present. Small molecules or polymer intercalations could be employed to functionalize it, and graphene oxide has employed made great progress in this area. It's been used in desalination, drug delivery systems, separation of oil and water, catalysis, solar cells, energy storage and healthcare [28-35] etc. On the other hand, single-component graphene has a number of drawbacks, including limited electrochemical activity, quick agglomeration, and complicated manufacturing, all of which limit graphene's application. As result, functional modification is required to expand the applications of graphene and graphene oxide. The use graphene oxide is crucial to expanding their application. The intrinsic structure of graphene and graphene oxide is further altered to achieve functionalization. We explore functional modification methods based on graphene and graphene oxide's intrinsic chemical bonds and functional groups. Firstly, following that, we'll go over the basics of graphene oxide, including its structure and properties.

Synthesized of Graphene Oxide:

In 2014, a modified Hummer's method was used to oxidize purified natural flake graphite and study the properties of graphene oxide. The graphene oxide was prepared by exfoliating graphite oxide in distilled water with ultrasonic waves. The structural and physiochemical properties of the products were investigated using scanning electron microscopy (SEM), X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and ultraviolet-visible spectroscopy (UV-vis). GO has a maximum absorption peak in the UV-vis spectra at 237 nm, because of the bonds between carbon atoms (C-C) that give the π-π* transition. Strong oxidizing agents like permanganate and chromates form oxygen functional groups on the surface of graphene like carbonyl, carboxyl, epoxy, and hydroxyl, but they also cause crystalline defects in the graphene sheet, according to FT-IR and Raman results. The XRD pattern revealed that graphene oxide had a peak at 12.02° with a layer spacing of 0.77 nm. The electrochemical behavior of a GO altered glassy carbon electrode was explored using the K3FeCN6 redox technique, and the results demonstrate that the electron transfer regulates the electrochemical behavior Leila Shahriary, Anjali A. Athawale [36]. In 2015, work describes the synthesis of graphene oxide (GO) used to exfoliate graphite flakes. There are two types of synthesis techniques, one is the common method and the other is the modified synthesis method. Graphene oxide was synthesized by the Hummer's method by oxidizing graphite sand, and the improved synthesis method incorporated the processes of oxidation and exfoliation of graphite sheets due to the heat treatment of the solution, and it was characterized by XRD, FT-IR spectroscopy, and SEM. FT-IR reveals the presence of O-H, COOH, and C=O as well as C=C bonds. The SEM picture also confirms the exfoliation of graphene sheets. As a result, the synthesized GO has a number of special and interesting properties that can be used in a range of applications. Paulchamy B, et al. [37]. The synthesis of GO in the cost-effective and efficient way remains a major problem. In 2016, By partially substituting KMnO4 with K2FeO4 and adjusting the amount of concentrated H2SO4, we were able to improve the NaNO3-free Hummer's methods. In comparison to the NaNO3-free Hummer's methods currently in use, this revised routine uses largely reduces the reactant consumption while maintaining a good yield. Various methods were used to characterize the acquired GO, an ultraviolet visible spectrophotometer was used to measure the optical absorption spectra of GO. FTIR spectroscopy was used to examine the chemical structure of GO, and use Al-Ka with X-ray photoelectron spectroscopy radiation and Raman spectroscopy. A thermal gravimetric analyzer with a 10 oC/min heating rate and a 50 mL/min Ar gas flow was used to determine the weight loss of samples. The phases were detected using a Cu-Ka X-ray diffractometer. FESEM and AFM were used to investigate the materials morphology. Huitao Yu, et al [38]. In 2017, GO made a modified Hummer's method using a reducing agent, hydrazine hydrate, that exfoliates graphite to produce rGO. A number of techniques, such as TGA, FTIR spectroscopy, Raman, FESEM, and XRD, were used to distinguish GO from rGO. According to XRD pattern, both GO and rGO have a crystalline structure. Because of the reduction, the functional oxygen groups (carbonyl, carboxyl, epoxy, and hydroxyl) present in GO were reduced, resulting in a lower d-spacing in rGO than in GO, and therefore the ratio in the intensity of the D and G bands (ID/IG) will increase. This means that p-conjugation is restored, which was obtained at two wavelengths, 532 and 785 nm, for the Raman excitation peaks for rGO. The bands are smaller. Under N2 flow, TGA thermograms for GO within thermal range 0-1000°C display higher overall weight loss, and the intensity of FTIR peaks for carbonyl, carboxyl, epoxy and hydroxyl groups was found to decrease significantly after reduction, FESEM image revealed that the surface of rGO is more wavy when compared with GO, this investigation is expected to be very useful for further development of GO/rGO-centered gas sensors to sense the exact gas concentrations, Neeru Sharma, et al [39]. In 2018 chemical reduction of graphene oxide (GO) to obtain reduced graphene oxide (rGO), for the first time, a mediated, easy, and relatively green method for preparing rGO in ethanol using artemisinin as a reducing agent is defined. The morphology and de-oxidation capability of resulting rGO were investigated using a transmission electron microscopy (TEM) and an atomic force microscopy (AFM) as well as photoelectron spectroscopy with X-rays (XPS), according to the findings, artemisinin can effective decrease GO into a few-layered rGO with a high carbon to oxygen ratio (11.7). The use of artemisinin as a technique for removing functional groups (OH, COOH and C=O) from GO nanosheets has been proposed. This technology has the advantages of being somewhat environmentally benign and having simple operating procedures, and it holds a lot of potential for mass production of rGO and other grapheme-based products, particularly biomaterials. Dandan Hou, et al [40]. In the year 2019, As a graphene source, over-oxidation of the carbon matrix made it difficult to build structure-property connections. A series of preparation protocols arose in an attempt to improve GO synthesis in order to achieve a less faulty material. Two alternative synthetic approaches for GO synthesis are shown to produce extremely similar GO forms with maintained graphene lattice. It is feasible to treat using sodium chlorate in nitric acid (as Brodie's procedure) or potassium permanganate in sulfuric acid (as in Hummer's method); The reaction conditions, on the other hand, must be closely monitored. Analytical variations between the samples with a retained carbon lattice contribute to the altered on-plane functionality. As a result, referring to preparation protocols as "Brodie's/Hummers' process" is inadequate. Patrick Feicht, et al. [41]. A modified Hummers' method was used to manufacture high-oxidation-degree GO particles in 2020. By altering the operation parameters, six different types of particles were created. Temperature, reactant ratios, and oxidation time are all variables to consider. The oxygen content reflects the degree of oxidation, CHNSO elemental analysis and X-rays photoelectron spectroscopy (XPS) were used to determine the atomic ratio of oxygen to carbon (O/C). Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were used to investigate the structural morphology of GO. The thermogravimetric technique (TGA) was used to assess thermal stability of particles. The produced GO samples displayed varied graphitic layer topologies, as shown by SEM images. Due to the difference in oxidation speed, the TEM images shown different stacking levels and clarity of GO flakes. Abedalkader Alkhouzaam, et al [42].

1-2. characterization of Graphene Oxide:

A variety of microscopic and spectroscopic techniques are used to characterize the morphology, quality, and structure of the graphene and the number of layers, as well as to detect the presence of defects. CHNSO elemental analysis was used to determine the oxygen content and the atomic ratio of oxygen to carbon (O/C). Fourier transform infrared (FTIR) spectroscopy, Raman spectroscopy and X-ray photoelectron spectroscopy (XPS), which are the most widely used characterization techniques. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were used to examine the structural morphology of graphene oxide. The thermogravimetric analysis (TGA) was used to investigate the particles' thermal stability.

1-3. Conclusion :-

Through the survey literature, the synthesis discovery of graphene oxides has revolutionized the nanotechnology and nanoelectronics industries to replace many materials like silicon, which have been utilized for decades and have now been expended into biomedicine, energy, nanorobotics, and other fields.

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