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Preparation Techniques of Graphene Oxide: A Review

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ABSTRACT: Graphene oxide (GO) is a versatile material derived from graphite, consisting of single-layer carbon atoms densely packed into a two-dimensional honeycomb lattice. It possesses a range of applications in various fields such as electronics, energy storage, and biomedical engineering due to its unique properties like high surface area, excellent mechanical strength, and good electrical conductivity. The preparation of graphene oxide typically involves several key steps, by which a bulk graphene oxide produces.

I. INTRODUCTION

Graphene, derived from graphite, is a two-dimensional sheet of carbon atoms arranged in a hexagonal crystal lattice. This lattice comprises two sub-lattices bonded with σ bonds, forming a robust structure [1]. The π orbitals in each carbon atom are partially filled beneath the plane [2]. The graphene sheets exhibit alternating single and double bonds between carbon atoms, contributing to its unparalleled strength, making it the most robust material known [3]. Additionally, graphene can be produced by fused benzene rings, varying in size from single or few rings to an infinite sheet. The lateral dimensions of single graphene sheets can range from centimeter to meter scales [4]. Despite its strength, graphene is incredibly lightweight, resembling an airborne material when separated into single sheets or a few layers [5]. Furthermore, graphene serves as a foundational element for other significant carbon allotropes, including 0D fullerene (C₆₀), 1D carbon nanotubes (CNTs), and 3D graphite [6], as illustrated in **Figure 1**.

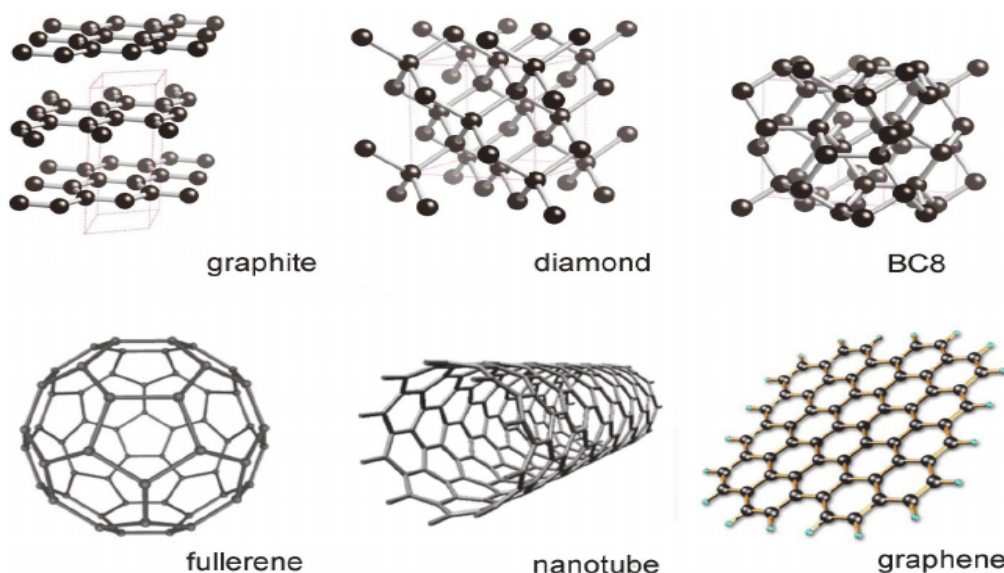


Figure 1: Geometrical nomenclature of carbon allotropes

Properties of graphene : Graphene stands out in comparison to carbon black or carbon nanotubes (CNTs) due to its significantly larger specific surface area (SSA) in theoretical terms. While carbon black typically exhibits an SSA smaller than 900 m²/g and carbon nanotubes (CNTs) have SSAs ranging from approximately 100 to 1000 m²/g, graphene surpasses them all with an impressive SSA value of around 2630 m²/g. This substantial SSA of graphene outshines even activated carbon, showcasing its superior potential in various technical applications [7].

Graphene, a unique 2D carbon allotrope, displays distinctive chemical reactivity on both sides of its sheet due to its structure. With the highest edge-to-atom ratio among allotropes, the atoms at the sheet's edges showcase special chemical reactivity. The introduction of defects amplifies its chemical reactivity, highlighting graphene's exceptional



potential in various technical applications [8]. Graphene is recognized as a zero-gap semiconductor because its valence band and conduction band intersect at points known as Dirac points. This unique characteristic distinguishes it from traditional semiconductors and contributes to its exceptional electronic properties[9]. Graphene exhibits remarkable electron mobility at room temperature, with reported values surpassing $15000 \text{ cm}^2 \text{ V}^{-1}\text{s}^{-1}$ [10]. Notably, even in the absence of scattering, graphene electrons can cover distances on the order of micrometers at room temperature, showcasing its exceptional conductivity and potential for high-performance electronic applications[11]. In a vacuum, graphene displays distinctive optical properties as an atomic monolayer. It exhibits an unexpectedly high opacity, absorbing approximately $\pi\alpha \approx 2.3\%$ of red light, with α representing the fine structure constant [12]. This heightened opacity is a consequence of the low-energy electronic structure of the graphene monolayer, where the conical bands of electrons and holes converge at the Dirac point. This unique electronic configuration contributes to graphene's exceptional optical behavior.

Graphene possesses an intrinsic tensile strength of 130 GPa and an impressive Young's modulus (stiffness) of 1 TPa (150,000,000 psi). Consequently, Graphene stands out as the strongest material ever tested, as indicated by various studies[13]. These exceptional mechanical properties underscore the remarkable strength and resilience of graphene, making it a forefront contender for advanced materials and structural applications[14]. In magnetic fields exceeding 10 Tesla, graphene exhibits the quantum Hall effect, unveiling additional intriguing features [1]. This phenomenon highlights the unique and complex quantum behavior of electrons in graphene under strong magnetic influences, providing a platform for exploring novel quantum states and potential applications in quantum technologies.

Graphene exhibits favorable wear behavior, with a low coefficient of friction typically ranging from 0.01 to 0.2 in dry sliding conditions. Its wear rate is significantly reduced, and the material demonstrates an improved load-carrying capacity[15]. Graphene contributes to the formation of protective tribofilms during wear, enhancing durability and reducing friction. Studies have explored temperature-dependent tribological properties, and wear mechanisms such as adhesion, abrasion, and fatigue are considered in quantitative analyses. The wear behavior of graphene varies based on factors like the form of graphene, substrate material, and testing conditions. Ongoing research continues to refine our understanding of graphene's wear properties, crucial for applications in lubrication, coatings, and composite materials[16].

Graphite nanoplatelets (GNP)

Graphite is a structured carbon allotrope characterized by alternating layers of sp^2 bonded carbon atoms arranged in trigonal planar sheets. Interaction between neighboring planes occurs through overlapping bonds in the unhybridized $2p$ carbon orbitals[10]. Each graphene sheet forms a hexagonal lattice, and weak Van der Waals forces bind these sheets together. These sheets stack to create platelets, forming aggregates that are 2-15 nm thick with diameters ranging from the sub-micron to $50 \mu\text{m}$. The carbon-carbon bond length within the basal planes measures 1.421 \AA , and the interlayer spacing is 3.354 \AA [17]. Graphitic carbons exhibit nonporous characteristics, offering a surface area of $20 \text{ m}^2 \text{ g}^{-1}$ and minimal hydrogen uptake at low temperatures [18].

Properties of Graphite Nanoplatelets

Graphite is identified as a semi-metal, featuring a complex band structure. Notably, it possesses a relatively low concentration of charge carriers, approximately 10 per carbon atom [19]. This characteristic is indicative of the unique electronic properties associated with graphite, contributing to its behavior as a semi-metallic material. Graphite serves as an electric conductor, primarily attributed to the high aspect ratio and expansive surface area of graphite nanoplatelets (GNPs). This configuration facilitates the establishment of an electrically conductive network at lower filler concentrations compared to conventional carbon black (CB) [20]. The ability of graphite to conduct electricity arises from the extensive electron delocalization within the carbon layers, a phenomenon known as aromaticity. This characteristic allows the valence electrons to move freely, enabling the effective conduction of electricity.

GNPs (graphite nanoplatelets) and graphite powder hold significant value in industrial applications, particularly for their self-lubricating and dry lubricating properties. The widely accepted belief attributes the lubricating characteristics of graphite to the loose inter-lamellar coupling between sheets in its structure [21]. This property makes both GNPs and graphite powder desirable choices for applications where effective and low-friction lubrication is essential in industrial settings. Graphite is highly anisotropic and diamagnetic, resulting in its ability to levitate in mid-air above a strong magnetic field. When produced in a fluidized bed at temperatures ranging from 1000 to 1300°C , it becomes isotropic and turbostratic, and is commonly utilized in blood-contacting devices such as mechanical heart valves. In this context,



it is referred to as pyrolytic carbon [22]. The unique properties of pyrolytic carbon make it suitable for applications requiring both biocompatibility and mechanical reliability in medical devices.

Graphene oxide

Graphene oxide (GO) is a two-dimensional carbon sheet with oxygenated functional groups, first synthesized by B.C. Brodie in 1859. It has gained recent popularity in the scientific community due to its unique properties, becoming a focal point of research and applications in the past five years.

As a crucial precursor to graphene, which earned Andre Geim and Konstantin Novoselov the Nobel Prize in Physics in 2010, graphene oxide (GO) holds scientific importance. Not only does GO serve as a foundational form of oxidized carbon, but it also boasts technological significance as a platform for diverse derivatives and composites. These derivatives and composites have showcased various intriguing applications, contributing to the broader scientific and technological landscape [23].

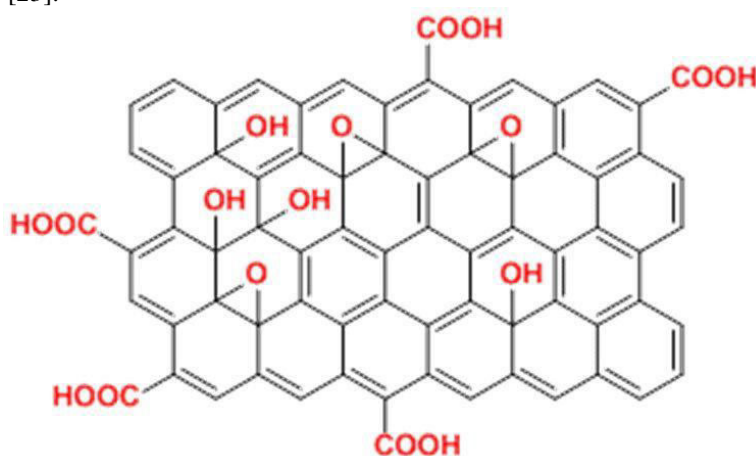


Figure 2: Structure of the graphene oxide molecule.

In **Figure 2**, it's essential to note that graphene oxide (GO) is not a naturally occurring compound; its origin lies in the chemical treatments of graphite. Initially synthesized by the British chemist Brodie, he referred to it as graphitic acid or graphite oxide [24]. Subsequently, following the advent of graphene research in 2004, the nomenclature shifted to graphene oxide. The synthesis of GO has undergone several modifications, involving diverse chemicals like potassium permanganate, concentrated sulfuric acid [25], and phosphoric acid [26].

Properties of Graphene Oxide

Functional Groups: The exact quantity of oxygen-containing functional groups on graphene oxide can vary, but it's typically expressed as a percentage of oxygen content by weight. For example, the oxygen content in graphene oxide might range from 20% to 40% [27].

Hydrophilicity: Hydrophilicity can be measured by contact angle, representing the angle formed between a water droplet and the surface of graphene oxide. Lower contact angles indicate higher hydrophilicity. Graphene oxide might exhibit contact angles ranging from around 30 to 70 degrees [28].

Electrical Conductivity: The electrical conductivity of graphene oxide is lower than that of pristine graphene. The sheet resistance of graphene oxide might be in the range of several hundred to several thousand ohms per square, depending on factors like synthesis methods and reduction treatments[21].

Mechanical Properties: Mechanical properties, such as Young's modulus and tensile strength, can vary. The Young's modulus of graphene oxide might range from 100 to 200 GPa, while tensile strength might range from 50 to 150 MPa[28].

Optical Properties: Optical properties can be quantified through parameters like absorbance and fluorescence intensity[29]. The absorbance spectrum of graphene oxide typically shows peaks in the UV and visible range, while fluorescence intensity depends on the specific functionalization and conditions[30].

Chemical Reactivity: Chemical reactivity is often characterized by the number of reactive sites or groups per unit area on the graphene oxide surface. This can be expressed in terms of moles of functional groups per gram of graphene oxide[31].



Biocompatibility: Biocompatibility can be assessed through various biological assays. For example, cell viability studies might quantify the percentage of viable cells when exposed to graphene oxide, and biocompatibility can be expressed as an IC50 value, indicating the concentration at which 50% cell viability is observed[32].

Gas Sensing: Gas sensing properties are typically measured by changes in electrical conductivity upon exposure to specific gases. The sensitivity can be quantified as the percentage change in conductivity for a given concentration of gas[33].

Methods for the oxidation of Graphite into Graphene Oxide

Five different methods for the preparation of GO in the chronological order may be given as Brodie Method, Staudenmaier Method, Hummers Method and Its Modification and Tour Method represent in **Figure 3**.

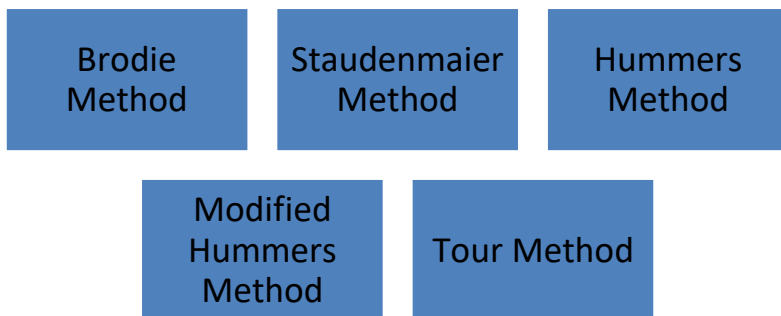


Figure 3: Five different methods for the preparation of GO

Brodie Method

In the 19th century, B.C. Brodie, a British chemist, played a pivotal role in the initial synthesis of graphene oxide (GO) during his exploration of graphite chemistry in 1859. During his investigations, Brodie introduced KClO₃ into a graphite slurry in fuming HNO₃, yielding a novel compound containing carbon, oxygen, and hydrogen. Subsequent steps involved washing the compound to remove the produced salt, drying it at 100°C, and subjecting it to further oxidation. Through three successive treatments, the appearance of the batch product underwent changes, ultimately resulting in a light-yellow substance resistant to additional oxidation treatments[34].

Brodie underscored the importance of a stepwise oxidation process, emphasizing that the final product, identified as C₁₁H₄O₅ through elemental analysis, could not be achieved through a single prolonged treatment. Each oxidation step required the restoration of the original condition. Despite displaying weak acidity and mild dispersibility in basic solutions, Brodie faced challenges in reflective goniometry characterization due to the small size, limited thickness, and imperfect structure of the product. Attempts to obtain GO salt through reactions with protochloride of copper and protochloride of tin were made. However, the observations and conclusions were constrained by the prevailing theories and characterization techniques of the time, leaving ample room for contemporary advancements and improvements [34].

Staudenmaier Method

L. Staudenmaier made notable advancements to Brodie's pioneering work in 1898[35].L. Staudenmaier introduced significant modifications to the graphene oxide (GO) synthesis process, bringing about two key changes:

Increased Acidity: Staudenmaier enhanced the acidity of the mixture by incorporating concentrated sulfuric acid. This adjustment aimed to optimize the oxidation conditions, influencing the chemical environment for the formation of graphene oxide.

Incremental Addition of Potassium Chlorate Solution: Another crucial modification involved the stepwise addition of multiple aliquots of potassium chlorate solution into the reaction mixture throughout the course of the reaction. This procedural change played a pivotal role in achieving a highly oxidized GO product, with a composition similar to Brodie's final product, all within a single reaction vessel. This innovation significantly streamlined the graphene oxide synthesis process.

These alterations introduced by Staudenmaier marked a notable improvement in the efficiency and simplicity of the GO synthesis method, providing a foundation for further advancements in the field[35].

Staudenmaier's approach, while advancing graphene oxide (GO) synthesis, had notable drawbacks. The method was time-consuming, with the addition of potassium chlorate extending over a week. Moreover, the evolved chlorine dioxide had to be eliminated using an inert gas, posing safety concerns due to the risk of explosions. Given these



challenges, there was a clear need for further modification or development of the oxidation process to enhance efficiency and safety. Subsequent investigations aimed to address these limitations and refine the synthesis methodology for graphene oxide.

Hummer's method and Its modification

Approximately six decades after Staudenmaier's approach, chemists Hummers and Offeman, at the Mellon Institution of Industrial Research, devised an alternative method for producing graphene oxide (GO) [34]. Their technique involved a water-free mixture comprising concentrated sulfuric acid, sodium nitrate, and potassium permanganate, maintained at temperatures below 45°C for graphite oxidation. The entire oxidation process, as described by them, concluded within two hours, yielding a final product with a higher degree of oxidation compared to Staudenmaier's method.

Despite Hummer's method, it was observed that the resulting product often featured an incompletely oxidized graphite core surrounded by graphene oxide (GO) shells. To enhance the degree of oxidation, a pre-expansion process became beneficial. In 1999, Kovtyukhova introduced a pivotal pretreatment involving heating graphite at 80°C with a mixture of concentrated H₂SO₄, K₂S₂O₈, and P₂O₅ for several hours, a practice widely adopted thereafter [35]. The pretreated mixture underwent dilution, filtration, washing, and drying before the actual Hummers oxidation step. Other reported modifications encompass adjustments in the quantity of potassium permanganate, among other factors. Presently, this modified Hummers method stands as the most prevalent approach for graphene oxide preparation, as depicted in **Figure 2**.

Tour Method

With the onset of the graphene research boom in 2004, graphene oxide (GO) swiftly became a focal point in carbon material investigations, prompting numerous publications exploring its structure, reduction, and applications. A noteworthy advancement came in 2010 from Professor Tour's group at Rice University, introducing a novel method that eliminated sodium nitrate, increased the potassium permanganate content, and introduced phosphoric acid into the reaction vessel [36]. This innovative approach involved reacting graphite with six equivalents of KMnO₄ in a 9:1 mixture of H₂SO₄/H₃PO₄. A significant advantage of this protocol is the absence of NaNO₃, mitigating the release of toxic gases like nitrogen dioxide and dinitrogen tetra-oxide during the reaction, rendering it more environmentally friendly [37]. Additionally, the use of phosphoric acid is believed to preserve more intact graphitic basal planes, resulting in a substantially higher final yield compared to Hummer's method [17].

Methods of reduction of Graphene Oxide into Reduced Graphene Oxide

Graphite oxide and graphene oxide both exhibit electrical insulation due to disruptions in their sp² bonding networks. The pivotal reaction for restoring electrical conductivity involves reducing graphene oxide, yielding products with various names like reduced graphene oxide (rGO), chemically reduced graphene oxide (CrGO), thermally reduced graphene oxide (TrGO), or simply graphene. While sharing chemical similarities with graphite oxide, graphene oxide differs structurally [38]. It transforms from a stacked structure to exfoliated monolayers or few-layered stacks during reduction. The surface functionality, especially in basic conditions, weakens platelet-platelet interactions due to hydrophilicity. The oxidation process itself causes the fragmentation of the graphitic structure into smaller fragments. Understanding the reduction mechanism is crucial for graphene oxide (GO) due to similarities between graphene oxide and reduced graphene oxide [39].

Chemical reduction

When graphene oxide (GO) is colloiddally dispersed using various chemicals, it serves as a precursor for reduction into reduced graphene oxide (rGO). Notably, hydrazine monohydrate was among the earliest and most commonly reported chemicals for this purpose [40]. Unlike many strong reductants with high reactivity in the presence of water, hydrazine monohydrate stands out, making it a preferred option for reducing aqueous dispersions of graphene oxide. However, the use of extremely potent reducing agents like lithium aluminum hydride (LAH) poses challenges due to side reactions with commonly used dispersing solvents, such as water. The primary objective of any reduction protocol is to generate graphene-like materials, akin to pristine graphene achieved through direct mechanical exfoliation (e.g., the "Scotch tape method") of individual graphite layers [41].

While the specific mechanism of hydrazine's reaction with graphene oxide, leading to its reduced counterpart, remains unclear, the product has undergone extensive characterization. Hydrazine monohydrate and a structurally similar species, di-amide, known for selectively reducing alkenes, are relatively mild reagents. This reduction typically involves the syn addition of H₂ across the alkene, coupled with the extrusion of nitrogen gas. This gentle process



preserves other functionalities, such as cyano and nitro groups. Various characterization techniques, including Raman spectroscopy, are employed to study the properties of both the resulting reduced material and the starting material. In Raman spectroscopy, the D band (associated with the order/disorder of the system) and the G band (indicative of the stacking structure) are dominant vibrational modes observed in graphitic structures. The ratio of the intensities of these bands (D/G) is commonly used to determine the number of layers and overall stacking behavior in a graphene sample, with higher D/G ratios indicating a higher degree of exfoliation/disorder [42]. Additionally, the electronic properties of the material are of significant interest.

Reduction through thermal mediation

Chemical reduction stands out as the most prevalent approach for reducing graphene oxide (GO). Research findings have been disclosed regarding the thermal exfoliation and reduction of GO [43]. Instead of employing a chemical reductant to remove oxide functionality, thermodynamically stable graphene oxide species can be created by directly heating GO in a furnace. The stacked structure undergoes exfoliation through the release of carbon dioxide upon heating GO to 1050°C. The high-temperature gas generates substantial pressure within the stacked layers, reaching up to 40 MPa at 300°C and 130 MPa at 1000°C, according to state equations [36]. Hamaker constant evaluation suggests that a pressure of only 2.5 MPa is required to separate two stacked graphene oxide platelets.

An interesting outcome of thermal exfoliation is the structural damage inflicted on the platelets due to the release of carbon dioxide [44]. About 30% of the GO mass is lost during exfoliation, resulting in vacancies and topological defects throughout the plane of the reduced graphene oxide platelet [45]. While these defects inevitably impact the electronic properties by reducing the ballistic transport path length and introducing scattering sites, bulk conductivities ranging from 1000 to 2300 S m⁻¹ were measured. This suggests an effective overall reduction and restoration of the planes and electronic structure. Despite potential effects on mechanical properties compared to chemically reduced samples, the substantial reduction achieved demonstrates the efficacy of thermal mediation in GO reduction.

Electrochemical reduction

An alternative approach to reduce graphene oxide involves the electrochemical elimination of oxygen functionalities. Previously, chemically reduced graphene oxide was coated with metallic nanoparticles through electrodeposition [46]. This method offers the advantage of avoiding the use of hazardous reductants (e.g., hydrazine) and eliminates the need to dispose of byproducts. The process involves depositing thin films of graphene oxide on various substrates (glass, plastic, indium tin oxide, etc.). Electrodes are then placed at opposite ends of the film, and linear sweep voltammetry is conducted in a sodium phosphate buffer. Reduction initiates at -0.60 V, reaching a maximum at -0.87 V. The reduction exhibits a rapid phase in the first 300 s, followed by a slower reduction rate up to 2000 s, and a subsequent decrease in background current levels up to 5000 s. Elemental analysis of the resulting material reveals a C:O ratio of 23.9:1, and the film exhibits a measured conductivity of approximately 8500 S m⁻¹ [47].

While this electrochemical reduction route appears highly effective and relatively mild in reducing existing oxide functionalities without the use of hazardous chemical reactants and byproducts, it has not been demonstrated on a large sample. The deposition of reduced graphene oxide onto electrodes may pose challenges for bulk electrochemical reduction on a preparative scale. The scalability of this method is a crucial consideration for its broader utilization in graphene-based applications.

Reduced Graphene Oxide (rGO)

The properties of reduced graphene oxide (rGO) fall between those of graphene oxide and graphene, exhibiting similarities to both materials. While rGO is moderately hydrophobic, allowing it to dissolve easily in various organic solvents, it also demonstrates limited solubility in water. Unlike graphene oxide, rGO presents challenges in its direct use as graphene. The term "reduced graphene oxide" encompasses a diverse range of materials produced from graphene oxide through deoxygenation or reduction processes. Despite the reduction, some oxygen groups persist in the structure of rGO, rendering it less soluble in polar solvents. The structural changes during the reduction process partially restore the aromatic structure, leading to the recovery of electrical conductivity in rGO [48]. **Figure 4** illustrates the structure of reduced graphene oxide.

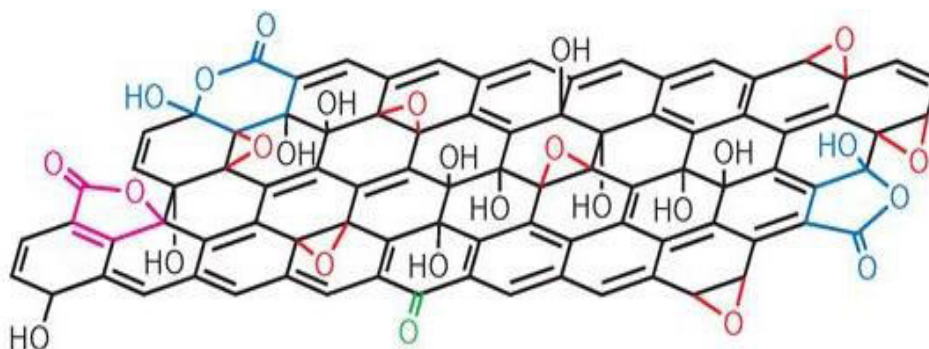


Figure 4: Illustrates the molecular composition of reduced graphene oxide (rGO)

II. CONCLUSION

There are various techniques of synthesis of graphene and reduced graphene. We have reviewed the different applications of GO/rGO. Despite the fact it's difficult to achieve the 100% reduction of graphene oxide into reduced graphene oxide. The bulk mass production in pure form is still challenge. The structure and the associate functional groups of graphite, GO and rGO represented in figure 1. The bulk mass production of graphene oxide and reduced graphene oxide must focus on two main points: The good understating of oxidation mechanism of graphite flaks. Control the graphite oxidation and functionality elimination during the reduction process of graphene oxide. The presence of functional groups is responsible for properties of graphene, they could be changes the properties of graphene oxide. For attainment of specific application, it required to tractable functionalization also is also important to obtained defect free graphene. There are various techniques also developed by various materials. But still for the bulk mass production graphite oxidation is favorable. So many research going on the oxidation and groups elimination with the analysis on the structure of graphene and the location of functional groups. May this research facilitate the applications of graphene-based sensors devices, transistors and photoelectronic devices etc.

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