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RESEARCH ARTICLE

SURVEY ON GRAPHENE AND GRAPHENE OXIDE: SYNTHESIS, STRUCTURAL PROPERTIES AND APPLICATIONS

***Tanmay Chattopadhyay**

Associate Professor, Department of Chemistry, Diamond Harbour Women's University, Diamond Harbour Road, Sarisha, South

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****Corresponding author:***
Tanmay Chattopadhyay

ABSTRACT

Graphene and graphene oxide have attracted significant attention over the past decade due to their exceptional and unique electrical, optical, mechanical, and chemical properties. This review focuses on the structure, characteristics, production methods, and applications of these materials. Initially, we provide a brief overview of the fundamental structure, production techniques, and properties of graphene and graphene oxide. Various methods, including chemical exfoliation, chemical vapor deposition, as well as top-down and bottom-up approaches, can be employed to synthesize these materials. Following this, we summarize the diverse applications of graphene and graphene oxide across various fields. Finally, we discuss the challenges that lie ahead for the development and utilization of graphene and graphene oxide. It is crucial to ensure that wastewater discharged during production contains minimal to no heavy metals, to mitigate any potential negative effects on the ecosystem.

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INTRODUCTION

Graphene is a carbon substance. In 1985, Robert Curl and his team produced C_{60} fullerene, marking a significant advancement in carbon research. (K. Geim *et al*. 2007; R. F. Curl *et al*. 1988). Four years later, Kratschmer confirmed the cage structure of C_{60} fullerene.[W. Kŕatschmer *et al*.1990] The discovery of carbon nanotubes by Nippon Electric Company Ltd. in 1991 further expanded the family of carbon materials. (Lijima, 1991) The pivotal moment for graphene came when Novoselov *et al*. (2004) successfully isolated it from bulk graphite, raising new questions about the nature of two-dimensional crystals. Graphene, consists of a single layer of sp^2 hybridized carbon atoms arranged in a hexagonal honeycomb lattice. With a thickness of just 0.34 nm, graphene is the thinnest and strongest known nanomaterial. (Slonczewski *et al*. 1958) Each carbon atom forms sigma bonds with three neighboring carbon atoms, while the remaining p electrons contribute to π bonds, which are oriented perpendicular to the graphene plane. This structure results in a stable arrangement, characterized by a short carbon-carbon bond length of 0.142 nm. (Zhang *et al*. 2005) Graphene's atomic structure ensures a strong bond between each carbon atom.

When subjected to external forces, the atomic surface bends and distorts, allowing it to counteract those forces without rearranging or losing alignment. (Novoselov *et al*. 2007) Consequently, the movement of electrons within graphene is largely unhindered by scattering from other atoms or lattice defects. (Novoselov *et al*. 2005; Wang *et al*. 2017) The remarkable lattice structure of graphene endows it with exceptional properties. Current methods for producing graphene include mechanical exfoliation, liquid-phase exfoliation, chemical vapor deposition, epitaxial growth, and redox methods. (Singh *et al*. 2011) Recent research has also focused on graphene quantum dots and carbon doped with other elements or compounds.(Mehta *et al*. 2019; Reddy *et al*., 2020; Liu *et al*., 2020) Researchers have identified that GO possesses a wealth of active functional groups containing oxygen, which can be utilized as catalytic active centers for both covalent and non-covalent modifications, depending on the specific requirements of various applications. The presence of oxygen-containing groups in graphene oxide increases the interlayer spacing, allowing for the intercalation of small molecules or polymers. Recently, there has been significant progress in the functionalization of graphene oxide, leading to its application across various industries, including desalination, drug delivery, oil-water separation, immobilized catalysis,

solar cells, energy storage, and healthcare (Nair *et al*. 2012; Liu *et al*. 2008; Feng *et al*. 2018; Li *et al*. 2013; J. Liu *et al*. 2012; Murat *et al*. 2017; Kumar *et al*., 2019; Shettia *et al*., 2019). However, the use of graphene is often limited by its inherent properties, such as low electrochemical activity, tendency to agglomerate, and difficulties in processing. To expand the applications of graphene and graphene oxide, functional modifications are essential. Enhancing the intrinsic structures of these materials lays the groundwork for effective functionalization. In this discussion, we will first outline the basic composition and characteristics of graphene and graphene oxide. Next, we will categorize their functionalization techniques into three groups based on these characteristics: 1) functional modifications involving covalent bonding; 2) modifications utilizing non-covalent interactions; and 3) element doping modifications. We will then provide a detailed summary of typical reaction processes, environments, and research methodologies for each type of modification. Finally, we will explore the potential of surface functionalization for graphene and graphene oxide.

Structure and Characteristics of Graphene and Graphene Oxide: The structure of graphene consists of planar hexagonal lattices. Each carbon atom has four valence electrons; three of these (2s, $2p_x$, and $2p_y$) form sp^2 hybrid orbitals, creating strong covalent bonds in the plane. The fourth electron remains in a p orbital, which allows for delocalized movement across the lattice. These unique structural and morphological features give both graphene and graphene oxide exceptional electrical, mechanical, and thermal properties. (Berger *et al*. 2004) Graphene exhibits high thermal conductivity, the Hall effect, tunneling effect, and bipolar electric field effect, with an ideal conductivity exceeding 1×10^6 S cm^{-1} and maximal electron mobility surpassing 2×10^5 cm^2 $(V\ s)^{-1}$ at room temperature, while its Young's modulus can reach up to 1100 GPa, transmittance for visible light is around 97.9%, and specific surface area can be as high as 2630 m^2 g^{-1}; in contrast, graphene oxide, a two-dimensional carbon compound with a single atomic layer and polydisperse sheet sizes, contains more oxygen-containing functional groups than graphene, which alters its structure and properties, as illustrated by the L-K model developed by Lerf and Kalinowski.

The L-K model, developed by Lerf and Kalinowski depicts the structural makeup of graphene oxide. Graphene oxide features carboxyl and carbonyl groups concentrated near the edges of the single layer, while hydroxyl and epoxy groups are randomly distributed throughout, resulting in regions of unoxidized benzene rings alongside oxidized six-membered aliphatic rings, with the sizes of these areas varying according to the level of oxidation and distribution. However, in favour of a set of presumptions, the L-K structural model disregards the effects of raw graphene, oxidants, and oxidation processes. The fundamental difference between graphene and graphene oxide is the presence of oxygen atoms bound to some carbons, according to several specialists who have conducted considerable study on the manufacturing and properties of G and GO, as shown in Fig. 1. Consequently, graphene has a hydrophobic nature, but graphene oxide has a hydrophilic nature and is readily dissolved in water. Additionally, the presence of both aromatic (sp^2) and aliphatic (sp^3) domains in graphene oxide increases the variety of interactions that can take place on its surface. A reducing agent can convert graphene oxide into graphene. However, because of the structural flaws introduced during the manufacture of graphene oxide, the generated graphene is unsuitable for electronic applications and mechanical reinforcement of polymers. However, this is the preferred method for significantly altering the surface characteristics of graphene materials by functionalization.

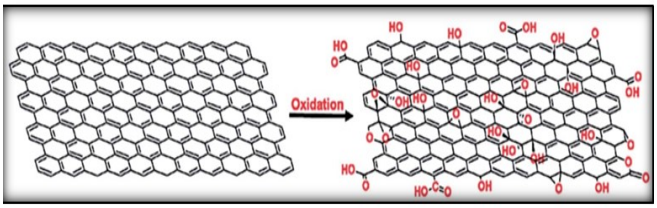

Fig 1. Graphene oxide formation by oxidation of graphene sheet

Synthetic methods of graphene and graphene oxide: The discussion of synthesis methods categorized into two sections: bottom-up approaches and top-down approaches.

Bottom-up approaches: Recent research has outlined the top-down and bottom-up synthesis processes for graphene-based materials, with the "bottom-up approach" referring to methods that construct materials from smaller components, akin to how a tree grows from a seed, where smaller atoms form molecules, leading to higher-order systems, as discussed in the following sections.

Epitaxial growth: A crystalline substrate is covered with layers of graphene using the synthetic process known as epitaxial growth. These many substrate types could include silicon carbide (SiC), hexagonal boron nitride (BN), Iridium (Ir), Ruthenium (Ru), Copper (Cu), Platinum (Pt), and more. A notable benefit of epitaxially grown graphene is that it can be patterned using a normal lithography technique (Sprinkle *et al*. 2009).

Chemical vapor deposition: Recent research has suggested that Chemical vapor deposition (CVD) may be a more practical synthesis approach for graphene. In this process, graphene is grown at high temperatures in the presence of a carbon-containing source gas on a metallic substrate (such as nickel or copper foil). Graphene grows when a transition metal substrate is exposed to a hydrocarbon gas at a high temperature. The Massachusetts Institute of Technology's first CVD paper from 2009describes attempts to synthesize an ultrathin graphene film with 1 to 10 layers using a nickel sheet at 900–1000°C and high-dilution hydrocarbon flow (Reina *et al*. 2009).

Hummer's Method: Graphite can be oxidized in an acidic atmosphere by using potent oxidizing agents as $KMnO_4$, $NaNO_3$, or $KClO_3$. In 1958, Hummer and colleagues showed how to use a reaction combination consisting of $KMnO_4$, $NaNO_3$, and H_2SO_4 in order to create a better graphene material (Choi *et al*. 2009). Since then, this reaction has been referred to as the modified Hummer's technique. Because GO has more oxygen-containing functional groups than pristine graphene, it can be chemically functionalized for use in a variety of applications.

Top-Down Approaches: In contrast to bottom-up tactics, top-down techniques include turning a higher order structure into a lower order entity.

Mechanical Exfoliation: The first graphene films were created using a mechanical exfoliation technique, also known as mechanical cleavage, in which layers of well-oriented pyrolytic graphite were repeatedly peeled off, revealing flakes of silicon that were about 3 nm thick and up to 10 m in size. Due to the very weak interlayer van der Waals force (Gupta *et al*. 2010), layer separation can be accomplished with ease using Scotch tape, SiO_2 stamps, palladium films. Some benefits of this synthetic technique include low cost, simplicity, and the capacity to generate vast amounts of graphene.

Chemical Exfoliation: In order to exfoliate the graphite and create graphene, a variety of reducing solvents have been used, including hydrazine hydrate, N-methyl-2-pyrrolidone, methane sulfonic acid and ionic solvents. A study by Fernández-Merino *et al*. (2010) examined the effects of hydrazine coupled with sodium borohydride, pyrogallol, and vitamin C. They sought a deoxygenation method for GO that would result in a well-dispersed product in aqueous and organic environments without precipitation after reduction.

Electrochemical Exfoliation: Graphite is used as a sacrificial electrode in the setup, along with several electrolytes such HBr, HCl, HNO_3, and H_2SO_4. H_2SO_4 created graphene with a large number of flaws while having the best exfoliation efficiency of these electrolytes in terms of time and thickness.In order to solve this issue, KOH was added to the H_2SO_4 solution to lessen the electrolyte's acidity and stop graphite from oxidizing as a result of H_2SO_4 alone. (Su *et al*. 2011) Recently, Gong *et al.* (2017) claimed that adding KOH at a low voltage of about 10 V caused the procedure to be too slow and ineffective.

Arc Discharge: High purity graphite electrodes are used in the arc discharge method to create graphene in the presence of buffer gases. He or H_2 have been utilized as buffer gases in the past. Due to the cessation of "dangling bonds" caused by the presence of H_2, H_2 buffer gas can hinder the creation of closed structures (Subrahmanyam *et al*. 2009). It has been demonstrated that adding helium to the buffer gas produces a material with a high crystallinity (Chen *et al*. 2012). Due of its ease of use, low cost, and most crucially the ability to generate graphene on a large scale with fewer faults, arc discharge has been employed as a synthetic process for graphene.

Applications: Graphene-based materials have recently been created for a variety of uses, including energy storage and conversion technologies, electrochemical sensors, absorption agents, and functional composite materials.

Suitable Composite Materials for Certain Functionally: Graphene-based materials can be employed as an additive component to create composite materials with the appropriate characteristics and performance thanks to their special features. In light of this, it is crucial for many applications to incorporate the qualities of graphene material-such as elasticity, porosity, strength, and conductivity-into the desired materials. Well-defined graphene matrices can offer a variety of composite applications high strength (Chen *et al*. 2013), super-hydrophobicity (Shin *et al*. 2010), high conductivity (Shin *et al*. 2010), flexibility (Chen *et al*. 2012), or reversible elongation (Chaubey *et al*. 2002).

Materials for Electrochemical Sensors: Electrochemical sensors can detect trace targets very effectively on graphene because of their huge surface area and strong conductivity (Shao *et al*. 2010). The rapid analyte transport made possible by large pore systems has improved mediated detection methods that can identify a variety of binding targets, including proteins and enzymes.

Absorbers: Comparing graphene sponges to other materials, they have far better organic liquid absorption capabilities with the added benefit of reusability. When exposed to gas, the functional groups on graphene oxide sheets might potentially shrink trapping and changing undesirable gases for use in industrial operations. Graphene sheets have also been shown to absorb some gases on a molecular level, allowing for resistive detection of low gas concentrations (Scheuermann *et al*. 2009).

Energy Storage and Conversion: Graphene materials are typically used as catalysts and catalyst supports for polymer electrolyte membranes (PEM) fuel cells; as cathode and anode materials for batteries like lithium batteries; as electrode materials for supercapacitors and solar cells, respectively; and as cathode and anode materials for batteries like PEM fuel cells (Wu *et al*. 2004).

Wastewater Remediation: Nanomaterials based on graphene oxide and graphene are both used to remove heavy metals. Many types of materials have been researched in relation to the creation of graphene oxide-based nanomaterials in order to eliminate the heavy metals stated above (Lim *et al*. 2018).

Future Challenge: The enhanced Hummers method is a widely used technique for synthesizing graphene, though it involves lengthy processes and minimal experimental complexity. To improve fabrication efficiency and affordability, it's essential to explore alternatives or reductions of specific chemicals. Given the increasing demand for clean water, effective methods for heavy metal removal from wastewater are crucial. Graphene oxide-based nanomaterials show significant promise in this area due to their accessibility and high surface area, which enhances removal effectiveness at lower costs. However, translating lab-scale results to industrial applications remains a challenge. Real-world testing of these nanomaterials in various water sources is necessary to evaluate their efficacy. Additionally, longer experimental periods and higher doses of nanomaterials should be used to ensure long-term effectiveness in wastewater treatment. Surface functionalization and the use of biomass waste as a resource could further enhance their adsorption capabilities while reducing costs. It's also vital to study the regeneration and reusability of these nanomaterials for sustainable industrial use. The successful implementation of graphene oxide-based nanomaterials in wastewater treatment could significantly benefit sectors such as metallurgy and support the establishment of more treatment plants to ensure safe water.

CONCLUSION

In summary, Top-down approaches, such as chemical exfoliation, are preferred for the production of graphene because they can result in graphene with fewer layers and a smaller size than Bottom-up methods. For instance, the enhanced Hummers method for chemical exfoliation of

graphene might produce nanoscale graphene that satisfies the need for manufacturing graphene oxide-based nanomaterials for heavy metal removal from wastewater. The manufacture, characteristics, and uses of materials based on graphene have been reviewed in this article. In summary, graphene and graphene oxide-based nanomaterials are known to be effective adsorbents connected with cutting-edge production techniques and technologies for the removal of heavy metals from wastewater to preserve the quality of the water.

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