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Eco-Synthesis and Characterization of Graphene Oxide Films for Chemicals Ensor Applications

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Abstract- ***The great demand for sustainable material in Nanotechnological industries has sparked the discovery of wonder material known as graphene and its derivatives (graphene oxide, reduced graphene oxide) because of its unique properties ranging from structural, electrical and optical properties. However, the conventional method has left chemical and high temperatures processes that are hazardous to human health and the environment. This research deals with an ecofriendly method of synthesis and deposition of graphene oxide (GO) using spin coating, achieving thin films with uniform structural, optical, and electrical properties. The Scanning Electron Microscope analysis at a magnification of 1µm, revealed a wrinkled morphology typical of GO, while Raman spectroscopy unveiled G band at ~1600 cm⁻¹ and D band at ~1350 cm⁻¹which confirmed the presence of graphitic domains and defects respectively. UV-V spectroscopy analysis reveals the absorption peak at 232 nm while the shoulder appears around 294 nm which veries the successful oxidation and exfoliation of graphite into GO, with well-defined optical characteristics. Hence, the linear I-V curve indicated consistent electrical behavior across the film. These findings demonstrate the potential of spin-coated GO films for applications in advanced materials and nanotechnology.***

Keywords: Eco-Synthesis, Graphene Oxide Film, Green Liquid-Phase Exfoliation

I. INTRODUCTION

The great demand for sustainable technology has sparked the discovery of novel materials and the modification of existing ones for application in a variety of spheres of human endeavour, particularly nanotechnology. Graphene, one of the novel materials was isolated successfully in 2004 by Manchester University academics, Novoselov and Geim. They received Nobel Prize awarded in 2010 for their pioneering work on graphene, Dresselhaus *et al.,*2010.

Graphene is a single layer of graphite with a 2D honeycomb-like lattice (sp^2 hybridization form) which possesses high-demanding properties like mechanical, electrical, thermal and optical properties in Nanotechnological industries, Worku & Ayele (2023). It is a semiconductor with zero band gap at the Dirac point, with very high electrical conductivity. Graphene and its derivatives (like graphene oxide, reduced graphene oxide and so no) can be synthesised from graphite and several methods such as flake fragmentation, Abbas *et al. (*2022) and growing Pei & Cheng, (2012). It was first separated from graphite by the mechanical stripping method in 2004, Mbayachi et al., (2021) and Singh *et al.* (2011). Graphene as an allotrope of the element carbon, is a planar sheet of carbon atoms arranged into hexagons (Veera (2019) and Katsnelson (2007)).

However, the development of sustainable and eco-friendly materials has gained significant attention in recent years, particularly in the field of nanotechnology. Hence, offers a promising alternative for graphene production, providing economic and environmental advantages. Graphene oxide (GO), a derivative of graphene, is an advanced two-dimensional material known for its exceptional electrical, thermal, and mechanical properties. Graphene oxide (GO) has a similar hexagonal carbon structure to graphene but also contains hydroxyl (–OH), alkoxy (C–O–C), carbonyl (C– – O), carboxylic acid (–COOH) and other oxygen-based functional groups, Pendolino & Armata (2017). One of the vital strengths of functional groups present in the graphene oxide is that its variation in the material can tune the electrical, mechanical, thermal, physical and other properties (Sharma *et al.,* 2021).

Graphene Oxide holds great promise for a variety of applications, including sensors, energy storage devices, and electronic components (Geim & Novoselov, 2007). However, traditional methods of synthesizing graphene and its derivatives often involve the use of hazardous chemicals such as Hydrochloric acid fuming (HCl), Potassium permanganate ($KMnO_4$), Concentrated sulfuric acid (H_2SO_4) and Hydrogen peroxide (H_2O_2) and energy-

intensive processes, raising environmental concerns ((Chen *et at.,* (2022) and Dreyer *et al.,* (2010)).

To address these challenges, this research has focused on developing green synthesis methods that minimize environmental impact while maintaining the desired properties of graphene oxide. One such approach is the liquid-phase exfoliation of graphite using environmentally friendly solvents such as acetone and deionized water, Avornyo & Chrysikopoulos (2024) and Hernandez *et al. (*2011). This method provides an efficient and scalable route for producing high-quality Graphene Oxide with controlled structural properties.

Spin coating is a widely used technique for depositing thin films of Graphene Oxide onto substrates such as glass slides, ensuring uniform distribution and optimal functional properties for sensor applications (Kim *et al.,* 2010). The characterization of these films is crucial for evaluating their structural integrity and performance. Techniques such as scanning electron microscopy (SEM), Raman spectrum, four-point probe measurements, and UV-visible spectroscopy provide comprehensive insights into the morphology, crystallinity, electrical conductivity, and optical properties of the synthesized Graphene Oxide films respectively (Zhou *et al.,* 2011).

This study aims to explore the eco-friendly synthesis and characterization of graphene oxide thin films for sensor applications. By optimizing the synthesis process and systematically analyzing the material properties, this research contributes to the growing body of knowledge on sustainable nanomaterial production and its practical applications.

II. MATERIALS AND METHODOLOGY

A. PREPARATION OF SOLVENT MIXTURE

Graphite source; Graphite flakes, ≥99% carbon, +50 mesh particle size (≥80%), natural purchased from Sigma Aldrich. Sonication power (e.g., watts) and centrifugation temperature are missing, hindering reproducibility

I mixed acetone and deionized water in a 7:3 ratio (acetone: deionized water), that is 70% acetone and 30% deionized water. This mixture can effectively disperse graphite while balancing exfoliation and stability. The solvent mixture was stirred thoroughly to ensure a homogeneous solution.

B. GRAPHITE DISPERSION

The graphite powder gotten from Graphite flakes, ≥99% carbon, +50 mesh particle size (≥80%), natural purchased from Sigma Aldrich was added to the acetone/deionized water mixture at a concentration of around 5 mg/mL. Hence, the mixture was stirred with a magnetic stirrer for 15 minutes to pre-disperse the graphite particles.

C. SONICATION PROCESS

The mixture was transferred to a suitable container that fits the ultrasonicator. The solution was then ultrasonicated in an ultrasonic bath for 2 hours, for moderate yield and good quality of graphene. It was ensured that the temperature of the mixture did not exceed 40°C during sonication using an ice bath, as higher temperatures can degrade the graphene quality. The study used 150W bath sonication at 40kHz for 30 munites

D. CENTRIFUGATION

After sonication, the mixture was centrifuged at 3000 rpm for about 40 minutes. This was done to separate larger graphite particles and unexfoliated flakes from the exfoliated graphene. The above condition was to divide the mixture into two (2) layers: supernatant and sediment. The supernatant (liquid part) will contain the exfoliated graphene, while the sediment will contain non-exfoliated graphite.

E. COLLECTION OF GRAPHENE OXIDE

To collect the graphene oxide, the supernatant was carefully decanted into a clean container without disturbing the sediment. This supernatant contains the few-layer or single-layer graphene oxide.

F. WASHING AND PURIFICATION

The deionized water was gradually added to the supernatant and the sample was washed several times to remove any residual acetone or impurities.

G. CLEANING THE GLASS SLIDE

The glass slides were washed thoroughly with soap and water to remove any grease or dirt that may alter the quality of the graphene. Hence, the slides were rinsed with acetone to remove organic residue contents. Subsequently, it was rinsed with deionized water to ensure all cleaning agents were removed.

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Finally, the glass slide was dried by placing it on a hot plate at a lot temperature (~60°C) to remove any remaining moisture.

H. SPIN-COATING

The cleaned glass slide was secured onto the spin-coater stage using a vacuum and a pipette was used to deposit a small droplet (approximately 200 µL) of the graphene dispersion onto the center of the glass slide. However, the spin-coater was started at a low speed (600 rpm) for 15 seconds to spread the liquid evenly. It was further increased the spin speed to a higher value (4000 rpm) and maintained this speed for about 50 seconds. The high-speed rotation helped to create a uniform thin film as the solvent evaporated rapidly.

I. ANNEALING THE FILM

After spin-coating, the glass slide was placed on a hot plate set to around 120°C for 7 minutes to ensure complete evaporation of the solvent and good film adhesion on the substrate (glass slides). The thickness was measured with a surface profilometer to be 22 µm and diameter to be 45mm

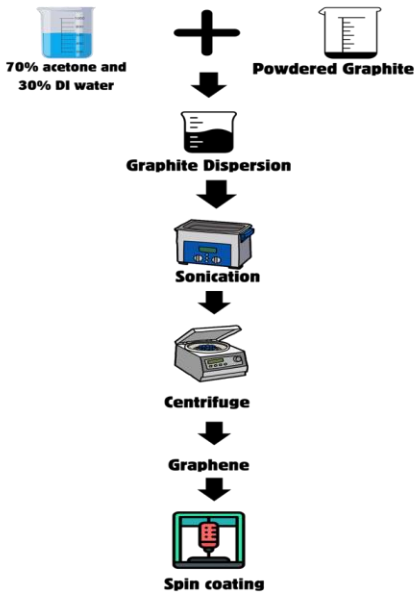

Figure 1: Schematic flow chart of the synthesis and deposition process

III. RESULTS AND DISCUSSION

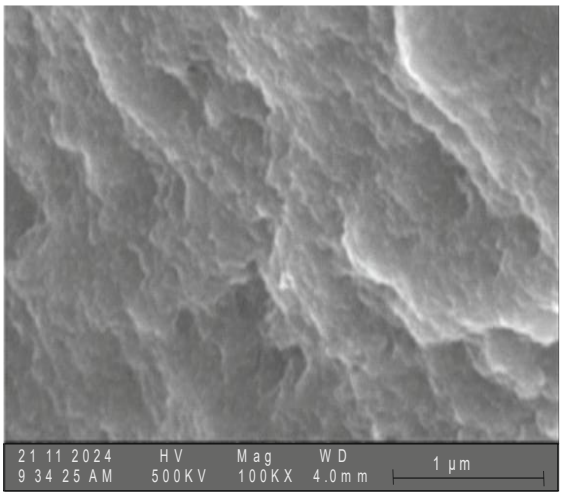

Figure 2 SEM images of Graphene Oxide film on a glass slide

X- At a magnification of 1 µm from Z SEM, the image provides a detailed view of the Graphene Oxide film structure, showcasing its microstructural arrangement at a fine scale. The image reveals a characteristic layered and wrinkled morphology, which is a well-known feature of graphene oxide films. These wrinkles are attributed to the drying and settling processes during spin coating, as well as the intrinsic structure of Graphene Oxide sheets, as previously reported in the literature (Park et al., 2009). Additionally, the continuity of the Graphene Oxide layer, as observed in the image, indicates a well-controlled spin-coating process, which is crucial for achieving uniform film deposition (Li et al., 2011). This morphology is particularly advantageous for applications in sensors, transparent conductive films, and electronic devices, where the unique properties of Graphene Oxide (GO) can be effectively utilized (Kim *et al.,* 2010).

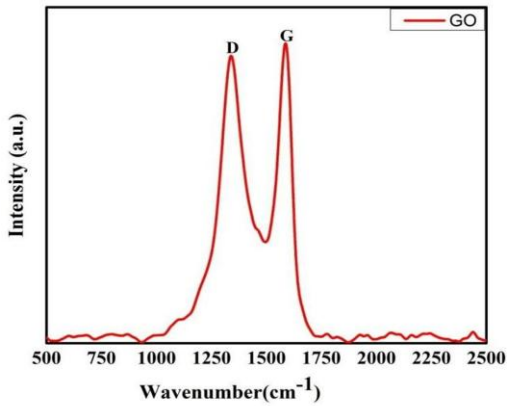

Figure 3: Raman spectrum for graphene oxide deposited on a Glass slide

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The D band at ~1350 cm^{-1} is attributed to the presence of defects and disorder in the carbon lattice. These defects arise from structural imperfections, oxygen-containing functional groups introduced during the oxidation process, and edge effects in graphene oxide (Ferrari *et al.,* 2015). The G band at ~1600 cm^{-1} corresponds to the in-plane vibrations of sp^2-hybridized carbon atoms within the graphene lattice. This peak is indicative of graphitic domains and is observed in both pristine graphene and graphene oxide (Dresselhaus *et al.,* 2010).

The distinct D and G peaks in the spectrum indicate a uniform deposition of graphene oxide on the glass substrate. The well-defined nature of these bands confirms that the spin-coating process has effectively distributed the Graphene Oxide layers across the surface (Zhou *et al.,* 2011).

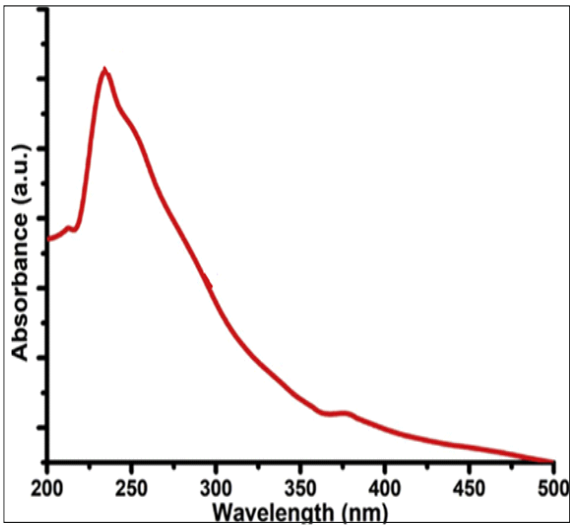

Figure 4: UV-Vis spectroscopy for graphene oxide deposited on glass slide

The prominent absorption peak observed at 232 nm in the UV-Vis spectrum corresponds to the π-π* transitions of aromatic C=C bonds in Graphene Oxide. This peak is a characteristic feature of the conjugated electronic structure within the graphitic domains of Graphene Oxide, reflecting the retention of sp^2 hybridized carbon atoms in the material's framework (Zhang *et al.,* 2010).

The shoulder appearing around 294 nm is attributed to n-π* transitions involving carbonyl (C=O) or other oxygen-containing functional groups. These functional groups are introduced during the oxidation process used to convert graphite into graphene oxide. Their presence disrupts the extended π-conjugation of the graphitic structure, introducing localized defect states within the material (Dreyer *et al.,* 2010).

The distinct and well-defined peaks in the spectrum indicate that the spin-coating process successfully produced a uniform and consistent deposition of graphene oxide on the glass substrate. This outcome underscores the effectiveness of spin coating in creating thin films with homogenous optical and structural properties suitable for further applications (Hassanzadeh *et al.,* 2014).

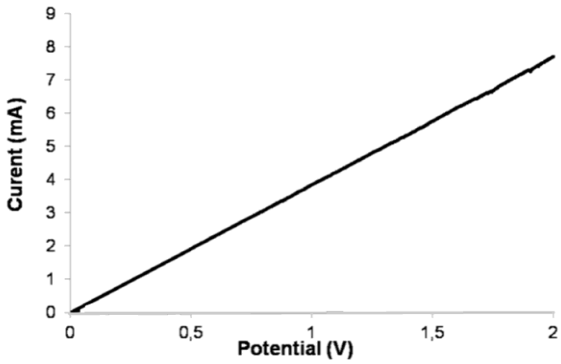

Figure 5: I-V curve showing sheet resistance for graphene oxide deposited on a Glass slide

The I-V curve exhibits a linear relationship between current (mA) and potential (V), indicative of ohmic behavior in Graphene Oxide thin films deposited on a glass slide via spin coating. Such behavior is characteristic of Graphene Oxide due to its insulating nature, which results from oxygen-containing functional groups that disrupt the conductive π-conjugated network within the material (Pei and Cheng, 2012). The linearity and smoothness of the I-V curve suggest that the spin-coating process effectively produced a uniform and defect-free Graphene Oxide film. Uniform deposition is essential for ensuring consistent electrical properties across the surface, which is crucial for applications requiring reliable conductivity and resistance (Dubey *et al.* 2017).

CONCLUSION

The study optimized the synthesis and deposition of Graphene Oxide using spin coating, achieving thin films with uniform structural, optical, and electrical properties. The SEM analysis revealed a wrinkled morphology typical of Graphene Oxide, while Raman spectroscopy confirmed the presence of graphitic domains and defects. and UV-Vis spectroscopy further verified the successful

oxidation and exfoliation of graphite into Graphene Oxide, with well-defined optical characteristics. The linear I-V curve indicated consistent electrical behaviour across the film. These findings demonstrate the potential of spin-coated Graphene Oxide films for applications in advanced materials and nanotechnology.

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