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REGULAR ARTICLE

Solvothermal Synthesis and Comprehensive Characterization of High-Quality Graphene Oxide

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This study presents the synthesis and characterization of a graphene oxide (GO) thin film prepared using a solvothermal method, optimized for thin-layered GO. Graphene oxide, obtained from graphite flake, is known for its versatility in applications such as electronics, energy storage, and catalysis, due to its layered structure, surface oxygen functionalities, and solubility. In this work, GO was synthesized through a solvothermal approach in N, N-dimethylformamide (DMF), enhancing the exfoliation of graphite oxide into thin GO sheets. The use of DMF facilitated effective layer separation, resulting in a few-layered GO with uniform thickness. The synthesized GO thin film was comprehensively characterized to determine its structural and functional properties. X-ray diffraction (XRD) confirmed the successful formation of GO through the observation of characteristic interlayer spacing. Fourier-transform infrared spectroscopy (FTIR) analysis revealed functional groups, such as hydroxyl and carbonyl groups, indicating GO's oxidized structure. Raman spectroscopy highlighted the defect density with distinct D and G bands, while scanning electron microscopy (SEM) provided insight into the film's morphology, showing well-dispersed, layered structures. Thermal gravimetric analysis (TGA) exhibited GO's thermal stability and decomposition profile, affirming the structural robustness of the synthesized thin film. This efficient solvothermal method shows promise for scalable GO thin-film production and assesses the environmental and economic feasibility of this approach to ensure sustainable application.

Keywords: Graphene oxide, Solvothermal synthesis, DMF, Characterization, Graphene-based materials.

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1. INTRODUCTION

Graphene oxide (GO) is an oxidized form of graphene that is rich in oxygen-containing functional groups, such as hydroxyl, epoxy, and carboxyl groups [1-5]. These functional groups modify the otherwise hydrophobic surface of graphene, making GO highly hydrophilic and easily dispersible in a range of solvents, including water. As a result, GO serves as an excellent precursor for functionalized graphene-based materials and offers unique properties for a range of applications. With the ability to disperse well in various solvents, GO provides opportunities for straightforward manipulation, film formation, and composite integration, broadening its use in fields such as electronics, catalysis, energy storage, and environmental remediation. The primary challenge in GO synthesis lies in achieving high yields of thin, defect-rich layers with controlled oxygen functionalities. While several methods exist for GO production, including the Hummers, Brodie, and Staudenmaier methods, the solvothermal approach offers unique advantages in terms of process control, product quality, and scalability [6-9]. The solvothermal method employs elevated temperatures and pressures in a solvent-filled, sealed environment to induce reactions that enable efficient exfoliation and modification of the starting materials. This process not only improves yield and quality but also offers tunable control over the structural and chemical properties of GO. The method's high temperatures and pressures facilitate the intercalation of solvent molecules between layers of graphite oxide, which leads to effective exfoliation and the formation of thin GO layers with desirable physical properties.

In this study, N, N-dimethylformamide (DMF) is employed as the solvent in the solvothermal synthesis of GO. DMF is a polar aprotic solvent, meaning it has a high polarity but lacks hydrogen atoms attached to electronegative atoms like oxygen or nitrogen [10]. This characteristic makes DMF highly effective in dispersing graphite oxide and facilitating intercalation between layers. In the solvothermal process, the use of DMF not only aids in exfoliating graphite oxide into graphene oxide but also prevents aggregation of GO layers due to its strong interaction with oxygenated groups on the GO surface [11]. Moreover, DMF's relatively high boiling point allows for elevated processing temperatures, which improves reaction kinetics and yields thin, high-

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quality GO films. The choice of DMF as a solvent also provides advantages for scalability, making the solvothermal approach feasible for large-scale production in industrial settings. Characterization of the synthesized GO is essential for understanding its properties and assessing its suitability for various applications. Structural analysis using X-ray diffraction (XRD) helps confirm the successful exfoliation of graphite oxide by identifying characteristic peaks associated with GO's interlayer spacing [12]. Fourier-transform infrared spectroscopy (FTIR) provides insights into the oxygen-containing functional groups on the GO surface [13], which play a key role in its reactivity and application potential. Raman spectroscopy reveals the defect density and disorder in GO's carbon lattice, a property that can significantly impact its electronic and mechanical behavior. Additionally, scanning electron microscopy (SEM) provides valuable information on the morphology of GO, showing whether a uniform layered structure has been achieved [14]. Finally, thermal gravimetric analysis (TGA) assesses GO's thermal stability, which is crucial for applications requiring resistance to high temperatures [15].

This study aims to demonstrate that the solvothermal method, using DMF as a solvent, is an efficient and scalable approach to synthesize high-quality graphene oxide thin films with controlled structure and functionality. By exploring the structural and functional properties of GO prepared through this method, we provide insights into its potential applications across various domains. The findings presented here contribute to a growing body of research focused on optimizing GO synthesis and tailoring its properties for specific applications, thereby advancing the development of graphene-based materials in industry.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in this study include high-purity graphite powder (≥ 99.9 %, Sigma-Aldrich), potassium permanganate ($KMnO_4$, Merck), sulfuric acid (H_2SO_4, Fisher Scientific), phosphoric acid (H_3PO_4, Sigma-Aldrich), hydrogen peroxide (H_2O_2, Merck), N, N-dimethylformamide (DMF, Sigma-Aldrich), and deionized water (Milli-Q, Merck Millipore).

2.2 Preparation of Graphite Oxide

Graphite oxide was synthesized following a modified Hummers' method [16-18]. Initially, 2 g of graphite powder was mixed with 80 ml of concentrated sulfuric acid and 20 ml of phosphoric acid in a round-bottom flask. The mixture was stirred at 50 °C to facilitate the oxidation reaction. Gradually, 10 g of $KMnO_4$ was added to the solution while maintaining the temperature below 50 °C to avoid overheating and excessive oxidation. The mixture was continuously stirred for 12 hours to ensure complete oxidation of the graphite. Following the reaction, the mixture was allowed to cool to room temperature. Subsequently, 50 ml of hydrogen peroxide was introduced to the solution to reduce any residual manganese ions present. The resulting graphite oxide was washed multiple times with deionized water until a neutral pH was attained. The product was collected through filtration and dried in a vacuum oven at 60 °C to yield a dry powder of graphite oxide.

2.3 Solvothermal Exfoliation to Obtain Graphene Oxide

To produce graphene oxide, 1 g of the prepared graphite oxide was dispersed in 50 ml of DMF using ultrasonication for 30 minutes to achieve a uniform dispersion. The dispersion was then transferred to a Teflon-lined autoclave and subjected to solvothermal conditions by heating at 180 °C for 12 hours. After the reaction period, the autoclave was allowed to cool to room temperature. The resultant product was filtered and washed with deionized water and ethanol to remove residual DMF and unreacted materials. The obtained graphene oxide powder was finally dried in a vacuum oven at 60 °C for 24 hours to ensure complete removal of moisture and solvents, resulting in a high-quality graphene oxide material ready for characterization.

2.4 Characterizations

In this study, the successful synthesis of graphene oxide (GO) was confirmed using a range of advanced characterization techniques. X-ray diffraction (XRD) was performed using a diffractometer equipped with a Cu Kα radiation source ($\lambda = 1.5406$ Å), operated at 40 kV and 30 mA, with a scan range of 5°-80° (2θ) and a step size of 0.02°. The Fourier transform infrared (FTIR) spectrum of GO film was recorded in transmittance mode with Perkin Elmer Spectrum 1000, measuring in the spectral range of 4000-400 cm^{-1} at a resolution of 2-4 cm^{-1}, using either the KBr pellet technique. Raman spectroscopy utilized a system with a 633 nm laser, operating below 10 mW to prevent sample damage, covering a spectral range of 400-4000 cm^{-1} with a resolution of ~1 cm^{-1}, and employing 50× or 100× objective lenses. Surface morphology was analyzed using scanning electron microscopy (SEM) with a field emission gun (FEG), at an acceleration voltage of 5-15 kV and a resolution of ~ 1 nm. Atomic force microscopy (AFM), operating in tapping with silicon probes of ~ 10 nm tip radius, provided nanoscale topographical details. Thermogravimetric analysis (TGA) was conducted using a thermal analyzer over a temperature range from room temperature to 1000 °C, at a heating rate of 10 °C/min, under a nitrogen or air atmosphere with a flow rate of 50-60 mL/min. Together, these techniques comprehensively characterized the structural, chemical, and thermal properties of the synthesized GO.

3. RESULT AND DISCUSSION

The solvothermal synthesis of graphene oxide (GO) using N, N-dimethylformamide (DMF) as the solvent has proven to be an effective approach for producing high-quality thin films with controlled structural and functional properties. Comprehensive characterization through multiple techniques, such as X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy, Raman spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and thermogravimetric analysis (TGA), has provided insights into the material's composition, structure, and stability, confirming the successful exfoliation and oxidation of graphite into GO.

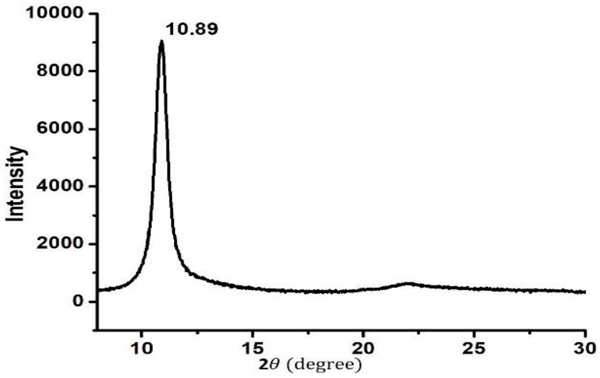

Fig. 1 – X-ray diffraction patterns of GO

The XRD pattern (Fig. 1) shows a characteristic peak at $2\theta = 10.89°$, corresponding to the (001) plane of GO, which is consistent with typical GO. This interlayer spacing signifies the successful oxidation and exfoliation of graphite oxide, in line with results from similar solvothermal approaches that use solvents such as N-methyl-2-pyrrolidone (NMP). Compared to conventional methods like Hummers' and Brodie's methods, the solvothermal technique provides better control over the exfoliation process, leading to more uniform GO layers with fewer defects. The solvothermal approach also allows for a scalable production process, making it more suitable for industrial applications, which is a key advantage over the traditional methods, which can be limited by long reaction times and high energy consumption.

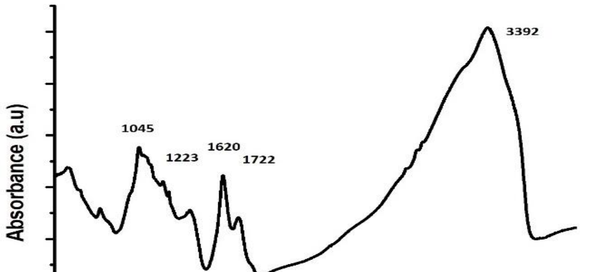

Fig. 2 – The Raman spectrum of GO

The FTIR spectroscopy further confirmed the oxidation of graphite, with distinct absorption bands corresponding to functional groups such as hydroxyl (–OH), epoxy (C–O), and carboxyl (C=O) at 3392 cm^{-1}, 1223 cm^{-1}, and 1722 cm^{-1}, respectively (Fig. 2). These oxygen-containing functional groups are crucial for improving the hydrophilicity of GO and enhancing its compatibility in aqueous environments, as seen in previous reports on GO synthesis via solvothermal routes. The introduction of these groups also makes GO an excellent candidate for a range of applications, such as in catalysis, energy storage, and sensors, where functionalization is often required to enhance performance. These findings align with the results from other solvothermal approaches, which also utilize DMF or similar solvents to obtain highly functionalized GO sheets with enhanced properties [13].

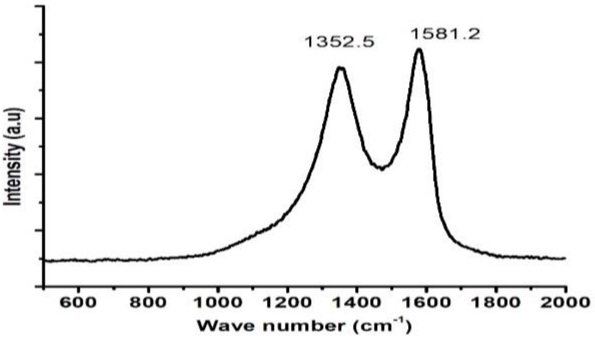

Fig. 3 – The Raman spectrum of GO

The presence of such functional groups not only provides insights into the chemical structure of GO but also highlights the extent of oxidation achieved. The carboxyl and hydroxyl groups contribute to its water dispersibility, while the epoxy and alkoxy groups confirm chemical modification at the basal plane and edges. These changes are essential for the functionalization of GO in various applications, including catalysis, energy storage, and biomedicine. Complementing this, the Raman spectrum (Fig. 3) reveals structural changes, further validating the successful synthesis of GO and the disruption of the pristine graphene lattice, crucial for its tailored properties and enhanced performance.

These functional groups enhance GO's hydrophilicity and confirm the degree of oxidation achieved [19]. Raman spectroscopy analysis revealed significant defect formation in the GO structure, with an ID/IG intensity ratio of 0.94. The D band, observed at 1352.5 cm^{-1}, represents defects or disorder, while the G band, appearing at 1581.2 cm^{-1}, corresponds to the in-plane vibration of sp^2 carbon atoms in Fig. 3. The ID/IG ratio suggests a relatively high degree of disorder, which can be advantageous for applications in composites and catalysis due to increased surface area and reactive site

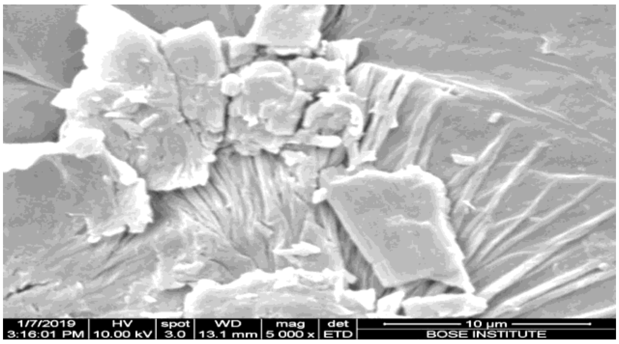

Fig. 4 – The SEM of GO

The morphological characteristics of graphene oxide (GO) were examined using SEM and AFM techniques. The SEM images (Fig. 4) revealed thin, layered sheets with a wrinkled texture, a hallmark of exfoliated GO nanosheets [14]. This distinctive morphology highlights the successful synthesis and structural integrity of the material. Complementary analysis through AFM (Fig. 5) confirmed the nanoscale thickness and uniformity of the GO sheets, providing further evidence of their well-defined structure. Together, these imaging techniques offer a comprehensive view of the surface topology and dimensional properties, underscoring the high quality of the prepared GO nanosheets for advanced applications.

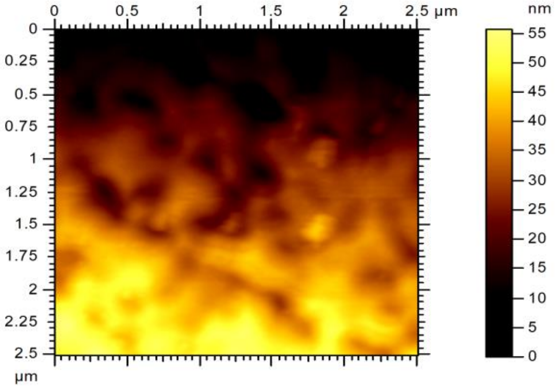

Fig. 5 – AFM images of GO

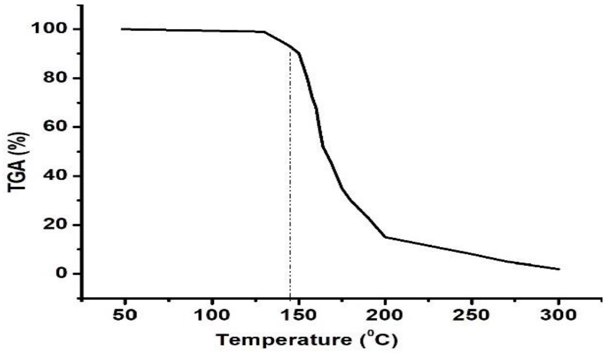

Fig. 6 – TGA images of GO

This was further supported by AFM, which confirmed the nanoscale thickness and the uniformity of the sheets (Fig. 5). The thermal stability of GO was evaluated using TGA under a nitrogen atmosphere [20]. The TGA curve (Fig. 6) exhibited gradual weight loss, with a prominent decomposition stage between 200-300 °C, corresponding to the breakdown of oxygen-containing functional groups. This thermal profile reflects the stability of GO and suggests its suitability for applications requiring moderate thermal resilience. The synthesized GO exhibited a well-defined structure with a high degree of oxidation, good thermal stability, and desirable morphological properties, making it a promising candidate for composite materials, catalysis, and other advanced applications.

4. CONCLUSION

This study successfully demonstrates the solvothermal synthesis of high-quality graphene oxide (GO) thin films using N, N-dimethylformamide (DMF) as a solvent, highlighting the method's scalability and efficiency. Comprehensive characterization through XRD, FTIR, Raman spectroscopy, SEM, and TGA confirms the production of a few-layered GO with uniform thickness, well-dispersed morphology, and robust thermal stability, establishing it as a versatile material for various applications. The unique structural and functional properties of the synthesized GO make it highly suitable for energy storage applications such as supercapacitors and lithium-ion batteries, where its layered structure and high surface area can enhance performance. Its oxygen functionalities also provide active sites for catalytic reactions in green organic transformations and environmental remediation, while its uniform thin film and defect density make it a promising candidate for flexible electronics, sensors, and conductive films. Furthermore, GO's hydrophilicity and large surface area can be utilized for adsorbing contaminants in wastewater treatment. Future work could focus on functionalization strategies to tailor GO properties for specific applications, scaling up this solvothermal method for industrial production, integrating GO into devices to validate practical performance, and assessing the environmental and economic feasibility of this approach to ensure sustainable and eco-friendly production.

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Сольвотермічний синтез та комплексна характеристика високоякісного оксиду графену

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У цьому дослідженні представлено синтез та характеристику тонкої плівки оксиду графену (GO), отриманої сольвотермічним методом, оптимізованим для тонкошарового GO. Оксид графену, отриманий з графітових лусочок, відомий своєю універсальністю в таких застосуваннях, як електроніка, накопичення енергії та каталіз, завдяки своїй шаруватій структурі, поверхневим кисневим функціональним групам та розчинності. У цій роботі GO було синтезовано сольвотермічним методом у N,N-диметилформаміді (DMF), що посилило розшаровування оксиду графіту на тонкі листи GO. Використання DMF сприяло ефективному розділенню шарів, що призвело до отримання кількашарового GO з рівномірною товщиною. Синтезовану тонку плівку GO було всебічно охарактеризовано для визначення її структурних та функціональних властивостей. Рентгенівська дифракція (XRD) підтвердила успішне формування GO шляхом спостереження характерних міжшарових відстаней. Аналіз за допомогою інфрачервоної спектроскопії з перетворенням Фур'є (FTIR) виявив функціональні групи, такі як гідроксильні та карбонільні групи, що вказує на окислену структуру GO. Раманівська спектроскопія виділила щільність дефектів з чіткими смугами D та G, тоді як скануюча електронна мікроскопія (СЕМ) дала уявлення про морфологію плівки, показавши добре дисперговані, шаруваті структури. Термічний гравіметричний аналіз (ТГА) продемонстрував термічну стабільність та профіль розкладання оксиду графену (GO), підтверджуючи структурну стійкість синтезованої тонкої плівки. Цей ефективний сольвотермічний метод є перспективним для масштабованого виробництва тонких плівок GO та оцінює екологічну та економічну доцільність цього підходу для забезпечення сталого застосування.

Ключові слова: Оксид графену, Сольвотермічний синтез, DMF, Матеріали на основі графену.