IJERST

International Journal of Engineering Research and Science & Technology

www.ijerst.org

ISSN : 2319-5991

Vol. 20 No. 4 (2024)

ijerst.editor@gmail.com
editor@ijerst.com

Int. J. Engg. Res. & Sci. & Tech. 2024

International Journal of Engineering Research and Science & Technology

ISSN 2319-5991 www.ijerst.com
Vol. 20, No.4, 2024

Research Paper

SYNTHESIS GRAPHENE OXIDE USING TOUR METHOD

Ms. Hetankshi Patel
Department of Physics,
Mehsana Urban Institute of Sciences, Ganpat University Ganpat Vidyanagar, Kherva (Mehsana),
Gujarat-384012

Abstract:

Due to its distinct structural, optical, and chemical characteristics, graphene oxide (GO), an oxidised derivative of graphene, has garnered a lot of interest for use in energy storage, medicinal devices, sensors, and composite materials. In this work, the Tour method—a modified and enhanced version of the traditional Hummers' method—was used to effectively synthesise graphene oxide from natural graphite. By using concentrated sulphuric acid and phosphoric acid as oxidising medium instead of sodium nitrate, the Tour technique improves oxidation efficiency, safety, and scalability. The principal oxidant, potassium permanganate, was employed, and carefully regulated reaction conditions were upheld to guarantee efficient oxidation while maintaining the structural integrity of graphene sheets. To create a stable GO powder, the synthesised graphene oxide was carefully cleaned, filtered, and dried. UV-visible (UV-Vis) spectroscopy was used to characterise the produced graphene oxide. The π–π* transition of aromatic C–C bonds were represented by a large absorption peak in the UV–Vi's absorption spectrum about 230 nm, while the n–π* transitions of C=O bonds were responsible for a shoulder near 300 nm. These spectrum characteristics verify that graphene oxide was successfully formed. This technique is appropriate for large-scale production and further uses of graphene oxide in advanced materials research due to its effectiveness and decreased environmental risks.

Keywords: Graphene oxide:; hemical oxidation; Graphite oxide; UV–Vi's spectroscopy; Wet chemical synthesis; Two-dimensional materials etc.

Received: 23-10-2024 Accepted: 02-12-2024 Published: 09-12-2024

1. INTRODUCTION:

One of nature's most adaptable elements, carbon may be found in a variety of allotropes, including diamond, graphite, fullerenes, carbon nanotubes, and graphene. Because of variations in atomic arrangement and bonding, every allotrope has unique physical, chemical, and electrical characteristics. Because of its extraordinary qualities and numerous prospective uses, graphene has become one of the most astonishing materials of the twenty-first century [1–5].

A two-dimensional (2D) single sheet of sp2-hybridized carbon atoms organised in a hexagonal honeycomb lattice is called graphene. Graphene has garnered significant scientific and technological attention since its initial experimental isolation in 2004 by mechanical exfoliation of graphite. It has exceptional qualities such a huge specific surface area, great optical transparency, superb mechanical strength, good thermal conductivity, and extremely high electrical conductivity. However, despite these exceptional qualities, cost, scalability, and process complexity constraints make it difficult to produce pure graphene on a wide scale [6–7].

Graphene oxide (GO) has drawn a lot of interest as a crucial graphene-based material to address these issues. A variety of oxygen-containing functional groups, including hydroxyl (–OH), epoxy (–O–), carbonyl (C=O), and carboxyl (–COOH) groups, are found on the basal plane and edges of the carbon sheets in graphene oxide, an oxidised version of graphene. Because of these functional groups, GO is hydrophilic and readily diffused in polar solvents like water. Consequently, reduced graphene oxide (rGO) and graphene-based composites may be made using graphene oxide as a flexible precursor [8–10].

To create graphene oxide from natural graphite through oxidation processes, several chemical techniques have been devised. The most popular techniques are the Tour method (2010), the Hummers method (1958), the Staudenmaier method (1898), and the Brodie approach (1859). Among them, the Hummers method has gained widespread use since it is more efficient than previous techniques and has a comparatively shorter reaction time. However, the conventional Hummers technique uses concentrated sulphuric acid and sodium nitrate, which can produce hazardous gases including nitrogen dioxide (NO_2) and dinitrogen tetroxide ($N2O_4$), raising safety and environmental issues [13–16].

Marcano et al. developed the Tour technique in 2010 as an enhanced and modified version of the Hummers method to overcome these drawbacks. The Tour technique uses concentrated sulphuric acid (H_2SO_4) and phosphoric acid (H_3PO_4) as the reaction media after sodium nitrate is removed. As a protective chelating agent, phosphoric acid lessens excessive oxidation and keeps the graphene sheets from suffering significant structural damage. This alteration produces graphene oxide with a greater degree of oxidation, a larger sheet size, and fewer structural flaws in addition to increasing the synthesis process' safety [17].

Because of its ease of use, affordability, increased output, and adaptability for large-scale production, the Tour technique is regarded as better. Better control over the oxidation process is made possible by the approach, which results in graphene oxide with improved structural integrity and repeatability. Additionally, it is more ecologically benign than previous approaches since there are no hazardous nitrogen-based byproducts [18].

To verify effective synthesis and comprehend the structural and optical characteristics of graphene oxide, characterisation is crucial. Ultraviolet–visible (UV–Vis) spectroscopy is a straightforward and efficient way to detect graphene oxide among many characterisation techniques. GO usually shows a shoulder about 300 nm, which is ascribed to n–π* transitions of C=O bonds, and a high absorption peak around 230 nm, which corresponds to π–π* transitions of aromatic C–C bonds. These spectrum characteristics are unmistakable markers of GO production and oxidation [19–20].

In this study, graphene oxide was created using a wet chemical oxidation process utilising the Tour technique. UV-Vi's spectroscopy was used to characterise the synthesised GO in order to verify its production. The goal of the study is to show that the Tour technique is a dependable, scalable, and cost-effective way to produce graphene oxide that is appropriate for advanced material applications.

2. Experimental Work and Characterization

2.1 Materials and Chemicals

Every chemical utilised in this study was of analytical reagent (AR) quality and didn't require any additional purification. Graphene oxide was synthesised using natural graphite powder as the carbon source. The oxidation and purification procedures used concentrated sulphuric acid (H_2SO_4), phosphoric acid (H_3PO_4), potassium permanganate ($KMnO_4$), hydrogen peroxide (H_2O_2), and distilled (DI) water. Because powerful acids and oxidising agents were used in the experiment, appropriate safety measures were taken.

2.2 Synthesis of Graphene Oxide by Tour Method

The Tour technique, a modified variant of the traditional Hummers method, was used to create graphene oxide. The first step in the procedure is to prepare an acid mixture using concentrated sulphuric acid and phosphoric acid in a 9:1 volumetric ratio. To create a homogenous reaction media, a determined amount of phosphoric acid and sulphuric acid was gradually combined while being continuously stirred. To prevent excessive oxidation and maintain the structural integrity of graphene sheets, phosphoric acid is an essential chelating agent.

To create a graphite intercalation compound (GIC), natural graphite powder was progressively added to the acid mixture while being vigorously stirred. To guarantee that the graphite was evenly distributed throughout the acidic medium, the combination was constantly agitated. Because the oxidation reaction is very exothermic, potassium permanganate ($KMnO_4$) was then gradually added in tiny amounts to the reaction mixture under carefully monitored conditions to prevent excessive heat generation. When $KMnO_4$ was added, the mixture's hue shifted from dark grey to greenish and finally dark brown, signifying the oxidation process.

To effectively oxidise the graphite layers, the reaction mixture was then heated and kept at a high temperature while being continuously stirred for a predetermined amount of time. Graphene oxide was created at this phase by adding oxygen-containing functional groups to the graphite structure's basal planes and edges. Hydrogen peroxide (H2O₂) was carefully added to the reaction mixture after distilled water was gradually added once the oxidation process was finished. The addition of hydrogen peroxide decreased the amount of leftover manganese species, changing the hue to a vivid yellow that is indicative of the synthesis of graphene oxide.

After then, the suspension was let to settle and cool. After discarding the liquid supernatant, the residue material was repeatedly cleaned with distilled water using centrifuge to get rid of any remaining acids, metal ions, and contaminants. Washing was done until the filtrate's pH was about neutral. To get graphene oxide in solid form, the purified graphene oxide was next filtered and dried in a moderately heated oven.

2.3 Purification and Drying Process

Purification is a critical step in the synthesis of graphene oxide to ensure the removal of unwanted by-products and residual reagents. In this work, repeated centrifugation and washing with distilled water were employed to achieve high purity. The wet graphene oxide cake obtained after filtration was dried in a hot air oven at approximately 90 °C for an extended duration. The drying process ensured the removal of moisture and resulted in fine graphene oxide powder suitable for further characterization.

Figure 1 Synthesis of Graphene Oxide by Tour Method

2.4 Characterization Techniques

Characterization of the synthesized graphene oxide is essential to confirm successful oxidation and to study its optical properties. Various analytical techniques can be employed for GO characterization; however, in the present study, ultraviolet–visible (UV–Vis) spectroscopy was used as a primary characterization tool due to its simplicity and effectiveness.

2.5 Ultraviolet–Visible (UV–Vis) Spectroscopy

A popular analytical method for examining the optical absorption behaviour of materials in the ultraviolet and visible portions of the electromagnetic spectrum is ultraviolet–visible spectroscopy. For detecting electrical transitions in carbon-based nanomaterials like graphene oxide, it is very helpful. To create a stable and consistent solution for UV-Vi's examination, a little quantity of synthesised graphene oxide was disseminated in distilled water using gentle sonication.

A UV-Vi spectrophotometer was used to record the graphene oxide dispersion's UV-Vi absorption spectra across an appropriate wavelength range. The π–π* electronic transitions of aromatic C–C bonds in the graphene framework are responsible for the prominent absorption peak that graphene oxide usually displays at 230 nm. Furthermore, n–π* transitions linked to carbonyl (C=O) functional groups are typically responsible for a shoulder or faint peak about 300 nm. The effective production of graphene oxide and the addition of functional groups containing oxygen are confirmed by the existence of these distinctive absorption characteristics.

Therefore, UV-Vi spectroscopy is a dependable and simple approach for confirming the production of graphene oxide produced using the Tour method.

3.0 Result and Discussion

3.1 UV–Visible Spectroscopic Analysis

Using ultraviolet–visible (UV–Vis) spectroscopy, the Tour method's effective production of graphene oxide (GO) was verified. UV-Vis spectroscopy is a commonly used method for the first characterisation of graphene oxide because it offers important details on electronic transitions and the existence of functional groups that include oxygen.

The produced graphene oxide dispersion's UV–Vis absorption spectra showed a prominent and powerful absorption peak at about 230 nm. The π–π* electronic transition of aromatic C–C bonds seen in the sp^2-hybridized carbon network is responsible for this peak. A distinctive aspect of graphene oxide is the presence of this peak, which signifies the partial restoration of conjugated π-electron systems upon oxidation.

A faint shoulder was seen about 300 nm in addition to the main absorption peak, which is indicative of n–π* transitions linked to carbonyl (C=O) and other oxygen-containing functional groups added during the oxidation process. The successful oxidation of graphite and the creation of graphene oxide sheets are further confirmed by the existence of this shoulder.

3.2 Effectiveness of the Tour Method

The observed UV–Vis spectral features demonstrate that the Tour method effectively introduces oxygen functional groups into the graphite structure while preserving the fundamental graphene framework. Compared to conventional oxidation methods, the Tour method provides better control over the reaction process due to the combined use of sulfuric acid and phosphoric acid. The phosphoric acid acts as a chelating agent, minimizing excessive oxidation and preventing severe structural damage to the graphene sheets.

A high degree of oxidation and consistent production of graphene oxide are indicated by the strong absorption peak at 230 nm and the distinct n–π* transition. These findings support earlier research, demonstrating that the Tour technique yields superior graphene oxide with enhanced structural integrity and a bigger sheet size.

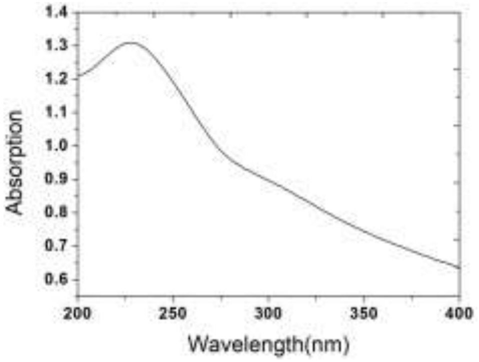

Figure 2 UV–Visible Spectroscopic Analysis

3.3 Discussion of Structural Implications

According to the UV-Vis data, graphite's π-conjugated structure was effectively disturbed by the oxidation process, resulting in the creation of graphene oxide with several oxygen functions. The hydrophilicity and dispersibility of GO in aqueous solutions are improved by these functional groups, which is crucial for additional processing and applications. The spectrum's optical behaviour is consistent with typical graphene oxide properties documented in the literature.

Overall, the findings demonstrate that the graphene oxide produced using the Tour approach is of high quality and appropriate for use in composite systems, energy storage devices, innovative materials, and sensors.

4.0 Conclusion

In this study, the Tour method—an enhanced and modified variant of the traditional Hummers method—was used to effectively synthesise graphene oxide from natural graphite. A combination of phosphoric and sulphuric acids was used in a wet chemical oxidation procedure to carry out the synthesis, which improved reaction efficiency and safety while reducing excessive oxidation and structural damage. The procedure became more ecologically friendly when sodium nitrate was removed since it further decreased the production of hazardous byproducts.

Using ultraviolet–visible (UV–Vis) spectroscopy, the production of graphene oxide was verified. The π–π^* transitions of aromatic C–C bonds are responsible for the distinctive absorption peak at about 230 nm, whereas the n–π^* transitions of functional groups containing oxygen are responsible for the shoulder at around 300 nm.

The findings show that the Tour technique is an easy, affordable, and scalable way to produce high-quality graphene oxide with acceptable structural integrity. A variety of applications, including as energy storage systems, sensors, biomedical devices, polymer composites, and advanced functional materials, can benefit from the features of the synthesised graphene oxide. All things considered, this work validates the Tour method's efficacy as a dependable pathway for the large-scale production of graphene oxide for scientific and commercial uses.

References

- C. Brodie, "On the atomic weight of graphite," *Philosophical Transactions of the Royal Society of London*, vol. 149, pp. 249–259, 1859.
- Eda and M. Chhowalla, "Chemically derived graphene oxide: Towards large-area thin-film electronics and optoelectronics," *Advanced Materials*, vol. 22, no. 22, pp. 2392–2415, 2010.
- J. M. Tour and D. V. Kosynkin, "*Graphene oxide synthesis: An overview*," Nature, vol. 458, pp. 872–876, 2012.
- Naga Charan Nandigama,"A Hybrid Big Data And Cloud-Based Machine Learning Framework For Financial Fraud Detection Using Value-At-Risk", Ijrar - International Journal of Research and Analytical Reviews (IJRAR), E-ISSN 2348-1269, P- ISSN 2349-5138, Volume.11, Issue 3, Page No pp.901-905, September 2024, Available at : http://www.ijrar.org/IJRAR24C3021.pdf
- Sankar Das, S. (2024). Transforming Data: Role of Data catalog in Effective Data Management. International Journal for Research Trends and Innovation, 9(12). https://doi.org/10.56975/ijrti.v9i12.207245.
- Lerf, H. He, M. Forster, and J. Klinowski, "Structure of graphite oxide revisited," *Journal of Physical Chemistry* B, vol. 102, no. 23, pp. 4477–4482, 1998.
- Naga Charan Nandigama. (2020). An integrated data engineering and data science architecture for scalable analytical warehousing and real-time decision systems. International Journal of Business Analytics and Research (IJBAR), Nov 2020, 10(1), 20-26.
- Q. Lai, S. Zhu, X. Luo, M. Zou, and S. Huang, "Ultraviolet–visible spectroscopy of graphene oxides," Analytical Methods, vol. 4, no. 8, pp. 2525–2532, 2012.
- R. Dreyer, S. Park, C. W. Bielawski, and R. S. Ruoff, "The chemistry of graphene oxide," *Chemical Society Reviews*, vol. 39, no. 1, pp. 228–240, 2010.
- S. Park and R. S. Ruoff, "Chemical methods for the production of graphenes," *Nature Nanotechnology*, vol. 4, no. 4, pp. 217–224, 2009.
- W. S. Hummers Jr. and R. E. Offeman, "Preparation of graphitic oxide," *Journal*

of the American Chemical Society, vol. 80, no. 6, p. 1339, 1958.