

Nano Graphene Oxide Synthesis, Characterization, and Application in Thermoplastics

Pradeep Uthale^{1*}, Sandeep Rai²

Abstract

The derivative graphene oxide (GO) is made of graphene, which is a layer of carbon atoms organized in a hexagonal lattice that is one atom thick. Graphene oxide possesses remarkable properties like high surface area, excellent mechanical strength, and electrical conductivity, making GO a material of interest for a wide range of applications. The synthesis of nano graphene oxide (nGO) involves oxidation that introduces oxygen functional groups onto the surface of the graphene sheet, making it dispersible in water and other solvents. This review provides an in-depth exploration of the various methods for synthesizing nano graphene oxide, the characterization techniques used to analyse its properties, and its potential applications in thermoplastics. The focus is on how nano graphene oxide can be integrated into thermoplastics to enhance their mechanical, electrical, and thermal properties, thus opening new opportunities for their use in various industries.

Keywords: Graphite, nano graphene oxide, thermoplastics, thermoplastics composites

INTRODUCTION

The remarkable properties of graphene led extensive research into its various derivatives, especially graphene oxide (GO), which has similar properties as that of Graphene but with increased functionality due to the presence of oxygen-containing groups. Nano Graphene Oxide (nGO), a nanomaterial form of GO, plays a key and crucial role in the development of advanced materials, particularly in polymer composites. The unique structure and properties of nGO, such as high surface area, flexibility, and the ability to interact with other materials, make it a potential candidate for incorporation into thermoplastic polymers matrix. Thermoplastics are a popular class of polymers that become soft and mouldable at elevated temperatures and solidify upon cooling.

It is anticipated that adding nGO to thermoplastics will greatly enhance their mechanical, thermal, and electrical characteristics. This review explores the various methods of synthesizing nano graphene oxide, the characterization techniques used to analyse nGO, and its applications particularly in thermoplastic materials.

*Author for Correspondence

Pradeep Uthale
E-mail: pradeep.uthale@gmail.com

¹General Manager R&D, Dyne Chemicals LLP, 3312/18, Chhatral Gujarat Industrial Development Corporation, Phase-IV, Taluka: Kalol, District: Gandhinagar, Gujarat, India

²Application Manger, Dyne Chemicals LLP, 3312/18, Chhatral Gujarat Industrial Development Corporation, Phase-IV, Taluka: Kalol, District: Gandhinagar, Gujarat, India

Received Date: February 15, 2025

Accepted Date: February 21, 2025

Published Date: March 04, 2025

Citation: Pradeep Uthale, Sandeep Rai. Nano Graphene Oxide Synthesis, Characterization, and Application in Thermoplastics. Journal of Modern Chemistry & Chemical Technology. 2025; 16(2): 1–7p.

NANO GRAPHENE OXIDE SYNTHESIS

Graphite undergoes oxidation to produce graphene oxide. The procedure entails applying functional groups that include oxygen, such as hydroxyl, carboxyl, and epoxy groups, to the edges and basal plane of graphene sheets. Several methods for the synthesis of nano graphene oxide are available, each having its own of advantages and challenges. Brief details of each methods are given as follows:

Hummers' Method

In 1859, Brodie first synthesized GO using potassium perchlorate (KClO_3) in fuming nitric acid (HNO_3) by oxidation of graphite [1]. However, the formation of toxic nitrogen dioxide gas and high explosive chlorine dioxide gas were big drawbacks of Brodie method. Subsequently, Hummers' method was developed in 1958 to overcome problems of toxic gas formation, and now is one of the most widely used techniques for producing graphene oxide from graphite [2]. It entails oxidizing graphite powder with a solution of potassium permanganate (KMnO_4) and sulfuric acid (H_2SO_4). Because the reaction is exothermic, it is necessary to carefully regulate the temperature to avoid producing too much heat and causing an accident. Chemical reaction scheme for synthesis of graphene oxide is given below in Figure 1.

Graphene oxide with a high degree of oxidation is produced by the Hummers' process. The resulting graphene oxide is typically brownish in colour and is highly dispersible in water, making it suitable for a wide range of applications (Figure 1).

Modified Hummers' Method [3]

The modified Hummers' method was developed to overcome environmental and safety issues associated with the original Hummers' method. This modified method reduces the use of hazardous reagents like potassium permanganate and is a more controlled and safer oxidation process.

Brodie's Method [1]

Brodie's method is another technique for synthesizing graphene oxide, which involves the use of potassium chlorate (KClO_3) in concentrated nitric acid (HNO_3) to oxidize graphite. This method yields a graphene oxide with fewer defects and a different oxygen functional group distribution compared to the Hummers' method.

Electrochemical Oxidation [4]

Electrochemical oxidation is a relatively recent method for synthesizing graphene oxide. In this process, graphite is subjected to an electrical current in an electrolyte solution, leading to the oxidation of graphite to graphene oxide. This method offers advantages such as high yield and the ability to control the degree of oxidation by adjusting the applied voltage.

Solvothermal Methods [5]

Solvothermal methods involve heating graphite in a solvent under high-pressure conditions. The structure and characteristics of the resultant graphene oxide can be affected by the solvent selection and temperature. This method is particularly effective in producing graphene oxide with specific characteristics tailored for certain applications.

CHARACTERIZATION OF NANO GRAPHENE OXIDE [6]

The characterization of nano graphene oxide is critical for understanding its properties and determining its suitability for various applications, including its incorporation into thermoplastics.

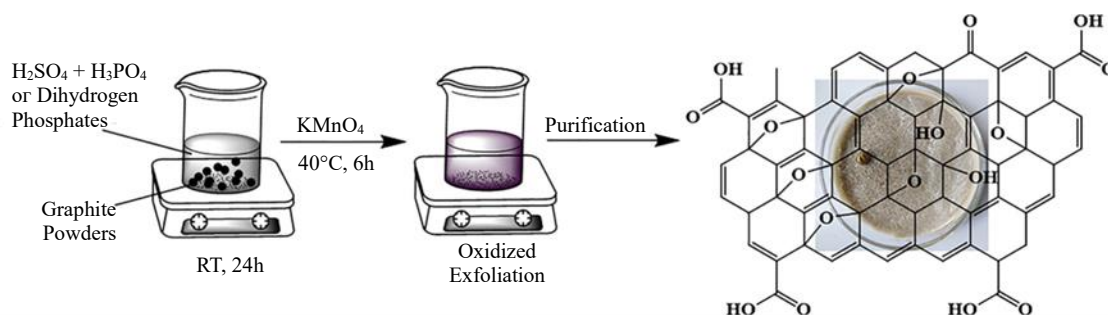


Figure 1. Hummers' method.

The morphology, structure, and characteristics of nGO are examined using a variety of methods. Key characterization techniques are mentioned below:

Scanning Electron Microscopy (SEM) [7]

Nanographene oxide surface morphology is frequently studied using scanning electron microscopy (SEM). High-resolution pictures of the nGO sheets may be obtained using SEM, displaying their dimensions, form, and surface properties. SEM analysis can also provide information about the dispersion of nGO in composite materials.

Transmission Electron Microscopy (TEM) [8]

Transmission Electron Microscopy (TEM) provides a more detailed view of the internal structure of nano graphene oxide at atomic resolution. TEM is useful for examining the number of layers of graphene oxide and the distribution of functional groups on the graphene surface.

Atomic Force Microscopy (AFM) [9]

Another effective method for determining the thickness and texture of individual graphene oxide sheets is Atomic Force Microscopy (AFM). AFM allows for the measurement of the nanoscale dimensions of nGO and the determination of its surface roughness.

X-ray Diffraction (XRD) [10]

The crystalline structure of nano graphene oxide is examined using X-ray diffraction (XRD). The XRD pattern of graphene oxide typically shows a prominent peak corresponding to the interlayer spacing of the graphene sheets, which provides information about the level of oxidation and the presence of functional groups.

Fourier Transform Infrared Spectroscopy (FTIR) [11]

Nanographene oxide's surface functional groups are examined using Fourier Transform Infrared Spectroscopy (FTIR). The FTIR spectrum typically shows peaks corresponding to hydroxyl (-OH), carboxyl (-COOH), and epoxy (-C-O-C-) groups, providing insights into the chemical composition of the material.

Raman Spectroscopy [12]

Raman spectroscopy is widely used to investigate the structural characteristics of graphene oxide. It provides information about the degree of disorder in the graphene sheets and the presence of different functional groups. The D and G bands in the Raman spectrum are commonly used to assess the structural integrity of nGO.

X-ray Photoelectron Spectroscopy (XPS) [13]

The elemental makeup and chemical state of the elements in nano graphene oxide are ascertained by photoelectron spectroscopy (XPS). XPS provides valuable information on the distribution of oxygenated groups, including hydroxyl, carboxyl, and epoxy groups, on the surface of nGO.

APPLICATIONS OF NANO GRAPHENE OXIDE IN THERMOPLASTICS [14–25]

Thermoplastic polymers are widely used in industries such as automotive, electronics, packaging, and construction due to their ease of processing and versatility. However, these materials often have limitations in terms of mechanical strength, thermal stability, and electrical conductivity. The incorporation of nano graphene oxide into thermoplastic matrices has shown promise in overcoming some of these limitations. Below are the primary applications of nGO in thermoplastics:

Mechanical Property Enhancement [26]

The mechanical characteristics of thermoplastics can be greatly enhanced by adding nanographene oxide. nGO acts as a reinforcing agent within the polymer matrix, increasing the tensile strength, modulus, and impact resistance of the composite material. Strong interactions with the polymer chains

are made possible by nGO's huge surface area and high aspect ratio, which improves load transmission and overall mechanical performance.

For instance, the addition of nGO to polycarbonate (PC) or polylactic acid (PLA) has been shown to improve their tensile strength and fracture toughness. The strong hydrogen bonding between the oxygenated groups on the nGO surface and the polymer matrix leads to better interfacial bonding, enhancing the overall mechanical properties.

Thermal Conductivity and Stability [27]

Nano graphene oxide can significantly improve the thermal conductivity and stability of thermoplastic materials. The high intrinsic thermal conductivity of graphene is retained in nGO, enabling the efficient transfer of heat through the polymer matrix. This is especially helpful in areas like electronics and automotive components where heat dissipation is essential.

Furthermore, nGO's oxygen functional groups contribute to thermoplastics' increased heat stability. The addition of nGO can reduce the degradation rate of the polymer when exposed to high temperatures, thus increasing the longevity of the material.

Electrical Conductivity [28]

Graphene oxide has semiconducting properties, and when incorporated into thermoplastics, it can impart electrical conductivity to otherwise insulating materials. This is particularly beneficial in applications where static dissipation, electromagnetic shielding, or conductive pathways are required.

By controlling the concentration and dispersion of nGO, thermoplastic composites can be engineered to achieve specific electrical conductivity levels. For example, nGO-filled polyvinyl alcohol (PVA) composites have been shown to exhibit improved electrical conductivity, making them suitable for use in sensors, electrodes, and other electronic devices.

Barrier Properties [29, 30]

Thermoplastics often suffer from poor barrier properties against gases and liquids. The incorporation of nano graphene oxide into the polymer matrix can enhance the barrier properties by reducing the permeability of the material to gases such as oxygen, carbon dioxide, and water vapor. In packaging applications, where enhanced barrier qualities prolong product shelf life, this is very helpful.

nGO-filled polymer films have been studied for their potential use in food packaging, where enhanced barrier properties can improve the preservation of food products by preventing the ingress of moisture and oxygen.

Flame Retardancy [31]

The addition of nGO to thermoplastic polymers has been shown to enhance their flame retardancy. Nano graphene oxide can promote the formation of a protective char layer during combustion, which helps in reducing the spread of flames and limiting the release of toxic gases. This is particularly useful in applications requiring fire-resistant materials, such as in electronics, automotive, and construction.

Studies have demonstrated that nGO can significantly reduce the flammability of thermoplastics like polypropylene (PP) and polystyrene (PS), making them more suitable for fire-sensitive applications.

Environmental Sustainability [32]

Thermoplastics derived from renewable resources, such as bioplastics, can benefit from the incorporation of nano graphene oxide to enhance their mechanical and thermal properties. nGO can also help in reducing the amount of plastic waste by improving the recyclability and durability of the material. Additionally, graphene oxide's ability to act as a filler and improve the overall performance of polymers can lead to a reduction in the use of other, more expensive or environmentally harmful materials.

CONCLUSION

A very adaptable substance, nano graphene oxide finds extensive use in thermoplastics. Through various synthesis methods, such as Hummers' method, modified Hummers' method, and electrochemical oxidation, nGO can be produced with varying degrees of oxidation, functionalization, and dispersion. Its characterization through techniques like SEM, TEM, XPS, and Raman spectroscopy provides valuable insights into its structure and properties.

When incorporated into thermoplastics, nano graphene oxide can significantly enhance the mechanical, thermal, electrical, and barrier properties of the polymer matrix. It can be used in a wide range of industries, including as construction, electronics, automotive, and packaging. The ability to tailor the properties of thermoplastic composites by adjusting the concentration and dispersion of nGO offers tremendous potential for developing advanced materials with improved performance.

As research continues into the synthesis, characterization, and application of nano graphene oxide, its role in the development of high-performance thermoplastics will likely expand, offering new possibilities for the design and manufacture of next-generation materials.

REFERENCES

1. Shirong Shuai, Yu Liu, Cong Zhao, Hongyu Zhu, Yang Li, Kanghong Zhou, Wei Ge, Jianyuan Hao. Improved synthesis of graphene oxide with controlled oxidation degree by using different dihydrogen phosphate as intercalators. *Chem Phys.* 2020; 539: 110938. ISSN 0301-0104, <https://doi.org/10.1016/j.chemphys.2020.110938>.
2. Songfeng Pei, Hui-Ming Cheng. The reduction of graphene oxide. *Carbon.* 2012; 50(9): 3210–3228. ISSN 0008-6223, <https://doi.org/10.1016/j.carbon.2011.11.010>. (<https://www.sciencedirect.com/science/article/pii/S0008622311008967>)
3. Zaaba NI, Foo KL, Hashim U, Tan SJ, Wei-Wen Liu, Voon CH. Synthesis of Graphene Oxide using Modified Hummers Method: Solvent Influence. *Procedia Eng.* 2017; 184: 469–477. ISSN 1877-7058, <https://doi.org/10.1016/j.proeng.2017.04.118>. (<https://www.sciencedirect.com/science/article/pii/S1877705817316235>)
4. Qingtao Yu, Luo Wei, Xiaoyong Yang, Chong Wang, Jikun Chen, Hongda Du, Wanci Shen, Feiyu Kang, Zheng-Hong Huang. Electrochemical synthesis of graphene oxide from graphite flakes exfoliated at room temperature. *Appl Surf Sci.* 2022; 598: 153788.
5. Scott Gilje, Kan Wang, Tung Vincent C, Kitty Cha, Hall Anthony S, Jabari Farrar, Rupal Varshneya, Yang Yang, Kaner Richard B. A One-Step, Solvothermal Reduction Method for Producing Reduced Graphene Oxide Dispersions in Organic Solvents Sergey Dubin. *ACS Nano.* 2010; 4(7): 3845–3852. DOI: 10.1021/nn100511a
6. Zhihao Zhang, Schniepp Hannes C, Adamson Douglas H. Characterization of graphene oxide: Variations in reported approaches. *Carbon.* 2019; 154: 510–521. ISSN 0008-6223, <https://doi.org/10.1016/j.carbon.2019.07.103>. (<https://www.sciencedirect.com/science/article/pii/S0008622319307961>)
7. Farah Abdullahi, Force Tefo Thema, Dikio Ezekiel. Electrochemical Detection of Hydrogen Peroxide Based on Graphene Oxide/Prussian Blue Modified Glassy Carbon Electrode. *Int J Electrochem Sci.* 2012; 7(6): 5069–5083. 10.1016/S1452-3981(23)19604-0.
8. Wu Shaoling, Zhao Xindong, Li Yan-Hui, Du Qiuju, Sun Jian-Kun, Wang Yonghao, Wang Xin, Xia Yanzhi, Wang Zonghua, Xia Linhua. Adsorption Properties of Doxorubicin Hydrochloride onto Graphene Oxide: Equilibrium, Kinetic and Thermodynamic Studies. *Materials.* 2013; 6(5): 2026–2042. 10.3390/ma6052026.
9. Obraztsova EA, Osadchy AV, Obraztsova ED, Lefrant S, Yaminsky IV. Statistical analysis of atomic force microscopy and Raman spectroscopy data for estimation of graphene layer numbers. *Phys Status Solidi B.* 2008 Oct; 245(10): 2055–9.
10. Sibirian Rikson, Sihotang Hotmaulina, Raja S, Supeno M, Simanjuntak Crystina. New Route to Synthesize of Graphene Nano Sheets. *Orient J Chem.* 2018; 34(1): 182–187. 10.13005/ojc/340120.

11. Çiplak Zafer, Karabudak Yildiz Nuray, Çalimli Ayla. Investigation of Graphene/Ag Nanocomposites Synthesis Parameters for Two Different Synthesis Methods. *Fuller Nanotub Carbon Nanostructures*. 2015; 23(5): 361–370. 10.1080/1536383X.2014.894025.
12. Childres I, Jauregui LA, Park W, Cao H, Chen YP. Raman spectroscopy of graphene and related materials. In: Jang JI, editor. *New Developments in Photon and Materials Research*. New York: NOVA Science Publishers, Inc.; 2013. p. 403–18.
13. Alessandro Kovtun, Derek Jones, Simone Dell’Elce, Emanuele Treossi, Andrea Liscio, Vincenzo Palermo. Accurate chemical analysis of oxygenated graphene-based materials using X-ray photoelectron spectroscopy. *Carbon*. 2019; 143: 268–275. ISSN 0008-6223, <https://doi.org/10.1016/j.carbon.2018.11.012>. (<https://www.sciencedirect.com/science/article/pii/S0008622318310297>)
14. Tiwari SK, Verma K, Saren P, Oraon R, De Adhikari A, Nayak GC, Kumar V. Manipulating selective dispersion of reduced graphene oxide in polycarbonate/nylon 66 based blend nanocomposites for improved thermo-mechanical properties. *RSC Adv*. 2017; 7(36): 22145–22155. [Google Scholar] [CrossRef]
15. Verdejo R, Bernal MM, Romasanta LJ, Lopez-Manchado MA. Graphene filled polymer nanocomposites. *J Mater Chem*. 2011; 21(10): 3301–3310. [Google Scholar] [CrossRef] [Green Version]
16. Martin-Gallego M, Bernal MM, Hernandez M, Verdejo R, Lopez-Manchado MA. Comparison of filler percolation and mechanical properties in graphene and carbon nanotubes filled epoxy nanocomposites. *Eur Polym J*. 2013; 49(6): 1347–1353. [Google Scholar] [CrossRef] [Green Version]
17. Shakir MF, Khan AN, Khan R, Javed S, Tariq A, Azeem M, Riaz A, Shafqat A, Cheema HM, Akram MA, et al. EMI shielding properties of polymer blends with inclusion of graphene nano platelets. *Results Phys*. 2019; 14: 102365. [Google Scholar] [CrossRef]
18. Yadav R, Tirumali M, Wang X, Naebe M, Kandasubramanian B. Polymer composite for antistatic application in aerospace. *Def Technol*. 2020; 16(1): 107–118. [Google Scholar] [CrossRef]
19. Tzounis L, Petousis M, Grammatikos S, Vidakis N. 3D Printed Thermoelectric Polyurethane/Multiwalled Carbon Nanotube Nanocomposites: A Novel Approach towards the Fabrication of Flexible and Stretchable Organic Thermoelectrics. *Materials*. 2020; 13(12): 2879. [Google Scholar] [CrossRef]
20. Franta I. Chapter 1: Introductory Part. In: Franta I, editor. *Elastomers and Rubber Compounding Materials*. Vol. 1. Amsterdam, The Netherlands: Elsevier; 1989; 19–30. ISSN 0922-5579. [Google Scholar]
21. Aguilar-Bolados H, Yazdani-Pedram M, Verdejo R. Thermal, electrical, and sensing properties of rubber nanocomposites. In: Valentini L, Lopez-Manchado M, editors. *High-Performance Elastomeric Materials Reinforced by Nano-Carbons*. Amsterdam, The Netherlands: Elsevier; 2020; 149–175. [Google Scholar] [CrossRef]
22. Nozaki S, Masuda S, Kamitani K, Kojio K, Takahara A, Kuwamura G, Hasegawa D, Moorthi K, Mita K, Yamasaki S. Superior Properties of Polyurethane Elastomers Synthesized with Aliphatic Diisocyanate Bearing a Symmetric Structure. *Macromolecules*. 2017; 50(3): 1008–1015. [Google Scholar] [CrossRef]
23. Aguilar Bolados H, Hernández-Santana M, Romasanta LJ, Yazdani-Pedram M, Quijada R, López-Manchado MA, Verdejo R. Electro-mechanical actuation performance of SEBS/PU blends. *Polymer*. 2019; 171: 25–33. [Google Scholar] [CrossRef]
24. Petrović ZS, Ferguson J. Polyurethane elastomers. *Prog Polym Sci*. 1991; 16(5): 695–836. [Google Scholar] [CrossRef]
25. Sami S, Yildirim E, Yurtsever M, Yurtsever E, Yilgor E, Yilgor I, Wilkes GL. Understanding the influence of hydrogen bonding and diisocyanate symmetry on the morphology and properties of segmented polyurethanes and polyureas: Computational and experimental study. *Polymer*. 2014; 55(18): 4563–4576. [Google Scholar] [CrossRef]

26. Maldonado-Magnere S, Yazdani-Pedram M, Aguilar-Bolados H, Quijada R. Thermally Reduced Graphene Oxide/Thermoplastic Polyurethane Nanocomposites: Mechanical and Barrier Properties. *Polymers*. 2021; 13(1): 85. <https://doi.org/10.3390/polym13010085>
27. Nazarychev VM. Enhanced Thermal Conductivity of Thermoplastic Polyimide Nanocomposites: Effect of Using Hexagonal Nanoparticles. *Polymers*. 2024; 16(23): 3231. <https://doi.org/10.3390/polym16233231>
28. Mostafizur Rahaman, Lalatendu Nayak, Ibbelwaleed A Hussein, Narayan Chandra Das. In *Polymer Nanocomposites Containing Graphene: Preparation, Properties, and Applications*. Woodhead Publishing Series in Composites Science and Engineering. Woodhead Publishing; 2022; 107–139. ISBN 9780128216392, <https://doi.org/10.1016/B978-0-12-821639-2.00025-2>. (<https://www.sciencedirect.com/science/article/pii/B9780128216392000252>).
29. Khanam PN, Ponnamma D, AL-Madeed MA. Electrical properties of graphene polymer nanocomposites. In: Sadasivuni K, Ponnamma D, Kim J, Thomas S, editors. *Graphene-Based Polymer Nanocomposites in Electronics*. Cham: Springer International Publishing; 2015. p. 25–47. doi: 10.1007/978-3-319-13875-6_2.
30. Jaweria Ashfaq, Iftikhar Ahmed Channa, Abdul Ghaffar Memon, Irfan Ali Chandio, Ali Dad Chandio, Muhammad Ali Shar, Mohamad S. Alsalhi, Sandhanasamy Devanesan. Enhancement of Thermal and Gas Barrier Properties of Graphene-Based Nanocomposite Films. *ACS Omega*. 2023; 8(44): 41054–41063. DOI: 10.1021/acsomega.3c02885,
31. Quanyi Liu, Yinlong Zhao, Shansong Gao, Xiong Yang, Rong Fan, Maoyong Zhi, Ming Fu. Recent advances in the flame retardancy role of graphene and its derivatives in epoxy resin materials. *Compos A: Appl Sci Manuf*. 2021; 149: 106539.
32. Hazel Lin, Tina Buerki-Thurnherr, Jasreen Kaur, Peter Wick, Marco Pelin, Aurelia Tubaro, Fabio Candotto Carniel, Mauro Tretlach, Emmanuel Flahaut, Daniel Iglesias, Ester Vázquez, Giada Cellot, Laura Ballerini, Valentina Castagnola, Fabio Benfenati, Andrea Armirotti, Antoine Sallustrau, Frédéric Taran, Mathilde Keck, Cyrill Bussy, Sandra Vranic, Kostas Kostarelos, Mona Connolly, José Maria Navas, Florence Mouchet, Laury Gauthier, James Baker, Blanca Suarez-Merino, Tomi Kanerva, Maurizio Prato, Bengt Fadeel, Alberto Bianco. *Environmental and Health Impacts of Graphene and Other Two-Dimensional Materials: A Graphene Flagship Perspective*. *ACS Nano*. 2024; 18(8): 6038–6094. DOI: 10.1021/acsnano.3c09699