AFFORDABLE GRAPHENE OXIDE COATINGS: SYNTHESIS AND EPD OPTIMIZATION

John Kinsner, Yeonjin Jung, PhD
Stephen F. Austin State University

STEPHEN F. AUSTIN STATE UNIVERSITY
THE UNIVERSITY OF TEXAS SYSTEM
NACOGDOCHES, TEXAS

Abstract

Graphene Oxide (GO) is a promising 2D nanomaterial with exceptional properties for advanced coating applications, including corrosion resistance and tribological performance. This research investigates the synthesis of GO via an improved modified Hummer's method and its deposition onto metallic substrates through optimized Electrophoretic Deposition (EPD). GO was synthesized from graphite powder using $KMnO_4$ and H_2SO_4, followed by controlled H_2O_2 addition and purification steps. Material characterization included AFM, SEM, and EDS. Results demonstrate successful GO synthesis with the formation of uniform, adherent coatings on stainless steel substrates. EDS analysis confirmed elemental composition with strong signals of carbon and oxygen, validating GO deposition. This methodology provides an accessible, cost-effective approach for producing functional GO coatings with potential applications in corrosion protection and tribological applications.

Figure 1: Graphited Powder.

Image Credit: www.neograf.com/products/powders-and-additives/grafplus-graphite-powders

Introduction

Graphene Oxide has emerged as a transformative nanomaterial in surface engineering applications due to its unique combination of high surface area (~2600 m^2/g theoretical), tunable surface chemistry, and exceptional barrier properties [1]. Unlike pristine graphene, GO possesses abundant oxygen-containing functional groups which enhance its dispersibility in aqueous solutions and enable strong adhesion to metal surfaces [2]. Moreover, GO can be synthesized through relatively low-cost, scalable chemical methods such as Hummers' method [3].

Recent research demonstrates that GO-based coatings provide exceptional tribological performance and corrosion resistance. In corrosion protection applications, GO protects metals from corrosion primarily by acting as an effective physical barrier; its layered nanosheet structure creates a highly tortuous path that blocks the diffusion of oxygen, water, and other corrosive agents, thus preventing them from reaching the metal surface [5, 6].

Electrophoretic Deposition (EPD) is a colloidal processing technique where charged particles suspended in a liquid medium migrate under the influence of an electric field and deposit onto an oppositely charged electrode [7]. EPD has emerged as a versatile technique for applying GO coatings to conductive substrates [8-10]. This research aims to develop and optimize a cost-effective methodology combining improved Hummer's method with systematic EPD parameter optimization to produce high-quality GO coatings suitable for tribological and corrosion protection applications.

Methods and Materials

Synthesis of Graphene Oxide, using Hummer's Method

Oxidation: After complete mixing, 36g of potassium permanganate ($KMnO_4$) was added slowly and carefully to avoid a sudden temperature rise and potential hazards due to overflow. The mixture was then stirred continuously for 72 hours to promote the thorough oxidation of the graphite structure into graphite oxide.

Termination: To terminate the oxidation reaction, 12 mL of 30% hydrogen peroxide (H_2O_2) was slowly added to the 2000mL beaker. The reaction produced visible bubbling, indicating the reduction of excess $KMnO_4$. The solution was further stirred with a magnetic stirrer for 30 minutes to ensure completion of the termination and uniform dispersion of the oxidized material.

Precipitation: 100 mL of deionized water was added, and the solution was stirred for an additional 5 minutes. The mixture was then left undisturbed for 3 hours to allow the solid precipitate to settle.

Purification: The precipitation was washed via a multi-step washing and purification process using DI water and a centrifuge.

Electrophoretic Deposition (EPD) Process

GO suspensions were prepared by dispersing dried GO powder in solvent medium, followed by magnetic stirring for 4 hours to ensure stable dispersion. A two-electrode configuration was applied for EPD, with the prepared substrates serving as both the working electrode (anode) and counter electrode (cathode). After deposition, samples were rinsed with deionized water and oven dried at 60°C for 3 hours.

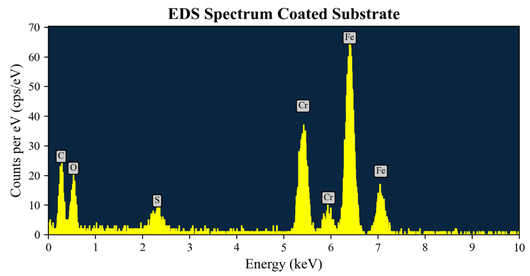


Figure 2: EDS spectrum of GO-coated Stainless-Steel Substrate

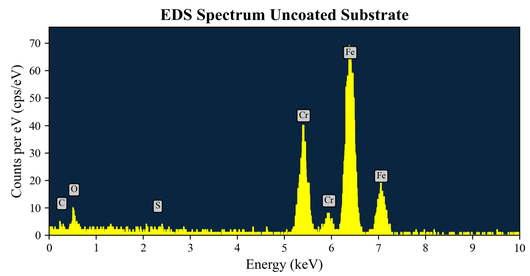
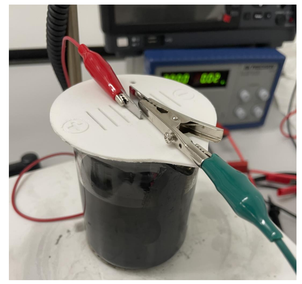


Figure 3: EDS spectrum of uncoated Stainless-Steel Substrate



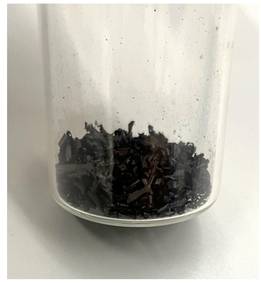


Figure 4: (First from left)

EPD Process using a 3D-printed cover. The cover includes slits for placing the parallel steel plates as well as a hole for the thermocouple.

Figure 5: (Second from left)

Synthesized GO powder in a container

Results and Discussion

Optimal Electrophoretic Deposition (EPD) conditions were determined to be 20 V applied voltage, 1.5 mg/mL GO concentration, 60 minutes, and 5mm electrode spacing. These parameters produced uniform and adherent coatings with minimal defects.

Higher voltages (>20 V) led to electrolysis and bubble formation, which disrupted coating uniformity. In contrast, lower voltages (10 V) resulted in insufficient driving force for complete GO migration. A higher GO concentration (1.5 mg/mL) yielded better performance due to the increased number of charged particles suspended in a liquid medium. While extended deposition times generally enhanced coating uniformity, excessively long durations (120 minutes) caused coating delamination due to excessive thickness and internal stress. The smaller electrode spacing (5 mm) proved more effective for coating formation than the larger spacing (10 mm).

As shown in Figure 2, EDS spectrum analysis confirms the successful EPD of GO onto the stainless-steel substrate. Distinct peaks corresponding to carbon and oxygen validate the presence of GO, as these elements are associated with the graphitic structure and oxygen-containing functional groups such as hydroxyl (–OH), carboxyl (–COOH), carbonyl (C=O), and epoxy (C–O–C) groups that define GO. A minor sulfur peak is also observed, which is attributed to residual sulfuric acid (H_2SO_4) used during the improved Hummers' method for GO synthesis. Overall, the EDS results confirm the successful formation of GO on the substrate surface, while also reflecting the substrate composition and synthesis-related residue.

Conclusions

This study demonstrated a cost-effective approach for synthesizing and depositing Graphene Oxide (GO) onto stainless steel substrates using an improved Hummer's method and optimized Electrophoretic Deposition (EPD) parameters. The optimal deposition conditions, 20 V applied voltage, 1.5 mg/mL GO concentration, 60 minutes deposition time, and 5 mm electrode spacing, produced uniform adherent coatings with minimal defects. Material characterization confirmed the successful synthesis of GO, with EDS indicating carbon and oxygen signals as GO presence. Minor sulfur traces validated the use of sulfuric acid during synthesis, while underlying substrate elements (Fe, Cr) confirmed the coating's thin profile.

Overall, the integration of low-cost chemical synthesis with controlled EPD processing provides an accessible and scalable method for fabricating GO-based surface coatings. These coatings exhibit potential for future applications in corrosion protection and tribological performance, supporting further development in surface engineering.

Contact

John Kinsner
Stephen F. Austin State University
kinsnerjp@jacks.sfasu.edu

Acknowledgements

This research was supported by the Department of Physics, Engineering, and Astronomy and the College of Science and Mathematics as part of the Summer Undergraduate Research Experience at Stephen F. Austin State University. We would also like to acknowledge the support of Dr. McDaniel, Dr. Friedfeld, and Mr. Watts, who assisted us throughout the project.

References

[1] Yadav, A., Panjikar, S., and Singh Raman, R. K., 2025, "Graphene-Based Impregnation into Polymeric Coating for Corrosion Resistance," Nanomaterials (Basel), 15(7).

[2] Muzyka, R., Drewniak, S., Pustelny, T., Sajdak, M., and Drewniak, L., 2021, "Characterization of Graphite Oxide and Reduced Graphene Oxide Obtained from Different Graphite Precursors and Oxidized by Different Methods Using Raman Spectroscopy Statistical Analysis," Materials (Basel), 14(4).

[3] Marcano, D. C., Kosynkin, D. V., Berlin, J. M., Sinitskii, A., Sun, Z. Z., Slesarev, A., Alemany, L. B., Lu, W., and Tour, J. M., 2010, "Improved Synthesis of Graphene Oxide," Acs Nano, 4(8), pp. 4806-4814.

[4] Chouhan, A., Mungse, H. P., and Khatri, O. P., 2020, "Surface chemistry of graphene and graphene oxide: A versatile route for their dispersion and tribological applications," Adv Colloid Interface Sci, 283, p. 102215.

[5] Fu, L., Sun, T., Shao, M., Xu, Z., Liao, K., Zhu, H., An, J., Jiang, L., and Gu, F., 2023, "Application of Modified Graphene Oxide in Corrosion Protection," ChemistrySelect, 8(28).

[6] Ding, R., Li, W., Wang, X., Gui, T., Li, B., Han, P., Tian, H., Liu, A., Wang, X., Liu, X., Gao, X., Wang, W., and Song, L., 2018, "A brief review of corrosion protective films and coatings based on graphene and graphene oxide," `Journal of Alloys and Compounds, 764, pp. 1039-1055.

[7] Chavez-Valdez, A., Shaffer, M. S., and Boccaccini, A. R., 2013, "Applications of graphene electrophoretic deposition. A review," J Phys Chem B, 117(6), pp. 1502-1515.

[8] Liu, Y., Chen, X., Li, J., and Luo, J., 2019, "Enhancement of friction performance enabled by a synergetic effect between graphene oxide and molybdenum disulfide," Carbon, 154, pp. 266-276.

[9] Liu, Y., Li, J., Yi, S., Ge, X., Chen, X., and Luo, J., 2020, "Enhancement of friction performance of fluorinated graphene and molybdenum disulfide coating by microdimple arrays," Carbon, 167, pp. 122-131.

[10] Mahmoudi, M., Raeissi, K., Karimzadeh, F., and Golozar, M. A., 2019, "A study on corrosion behavior of graphene oxide coating produced on stainless steel by electrophoretic deposition," Surface and Coatings Technology, 372, pp. 327-342.

© Poster Template by Genigraphics® 1.800.790.4001 www.genigraphics.com