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Synthesis and Characterization of Graphene Oxide Nanoparticles Using Graphite Dust

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Abstract: *Graphene oxide has numerous applications in several scientific fields including electrical, environmental, energy and other sectors. Given their significance, the present study was conducted to produce nanographene oxide from locally produced graphite dust. The production yielded 87% nanographene oxide through Hummer's method. The synthesized product showed stretching vibrations due to C-O and also displayed highly porous surfaces as distinct from those observed in the FTIR spectrum and SEM micrograph of the precursor respectively. The study adds to existing literature that nanographene oxide can be synthesized from graphite dust by Hummer's method.*

Keywords: *Graphite dust, nanographene oxide, synthesis, Hummer's method, FTIR, SEM*

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1.0 Introduction

Nanomaterials are known for their outstanding properties such as surface area, pore diameter, pore volume, optical properties, electrical properties, mechanical and other properties (Eddy *et al*., 2023a-b). The listed and other properties of nanoparticles have guaranteed their applications in several scientific areas including electronics, environment, agriculture, health, pharmaceutical, etc (Ogoko *et al*., 2023; Eddy *et al*., 2022a-b; Odoemelam *et al*., 2023). Among the most widely known non-metal oxide nanoparticles are graphene oxide nanoparticles, which a carbon-based nanoparticles (Alzubaidy *et* al., 2023; Din *et al*., 2023; Oukhrib *et al*., 2023). Graphene oxide is an oxidized carbon-based nanoparticle and is typically made up of a single-atom-thick layer of graphene sheets with carboxylic acid, epoxide and hydroxyl group in the plane (Uzhviyuk *et al*., 2023). Several advantages associated with the applications of graphene oxide have been reported including applications in electronics, environmental and biomedical sectors (Singh *et al*., 2023). The success associated with the broad application of graphene oxide is linked to its properties such as planar shape, high aspect ratio, good optical properties, electrical conductivity, mechanical and thermal stability (Afraid, 2023; Baragaño *et al*., 2020; Rhazouani *et al*., 2021). Because of their significant applications, graphene oxide production is ongoing at different scales. However, it has been established that nanoparticles have better performance efficiency than their classical materials. Therefore, better performance is expected from graphene oxide nanoparticles than the graphene oxide. Consequently, this study aims to synthesise graphene oxide nanoparticles from graphite dust and characterise the product using FTIR and SEM.

2.0 Materials and Methods

Graphite dust was supplied by Prof. Nnabuk Okon Eddy of the University of Nigeria, Nsukka. Hummer's method was adopted for the synthesis of the graphene oxide nanoparticles as reported elsewhere (Yu *et al*., 2016. The Avatar 330 FT-IR instrument was used for FTIR analysis while the SEM images were taken using a Concise FEGSEM 6100 Zeiss Ultra Plus (Germany) under an accelerating voltage of 20 kV

3.0 Results and Discussion

The chemistry of the Hummers' method associated with the synthesis of nanographene oxide is defined by the initial oxidation of graphite to graphene oxide through the oxidation process, which introduces molecular oxygen into the pure carbon graphene. The reaction occurs between the graphene and the concentrated sulfuric acid with the potassium permanganate and sodium nitrate acting as catalysts. According to Ji *et al*. (2009), the oxidation process involves intermediate formation of inert HSO_4 and further conversion to $C_x^+ - [HSO_4]^-$, which is oxidized into nanographene oxide (Ji *et al*., 2009). The process yielded 87 % of nano graphite oxide. The final product contained 47.02 % carbon, 27.97 % oxygen, 22.99% water, and 1.98 % ash with a carbon-to-oxygen ratio of 1.68. All of these results are comparable to those reported by others (Bolibok *et* al., 2021; Muthu *et al*., 2022; Tas and Cakmak, 2020; Wei *et al*., 2019) The FTIR spectrum of graphite (Fig. 1) reveals a broad peak, which appeared at 3446 cm^{-1} in the high-frequency area and is attributed to the stretching mode of O-H bond and it indicates the presence of hydroxyl groups in the synthesized nanographene oxide (Karteri *et al*., 2014).

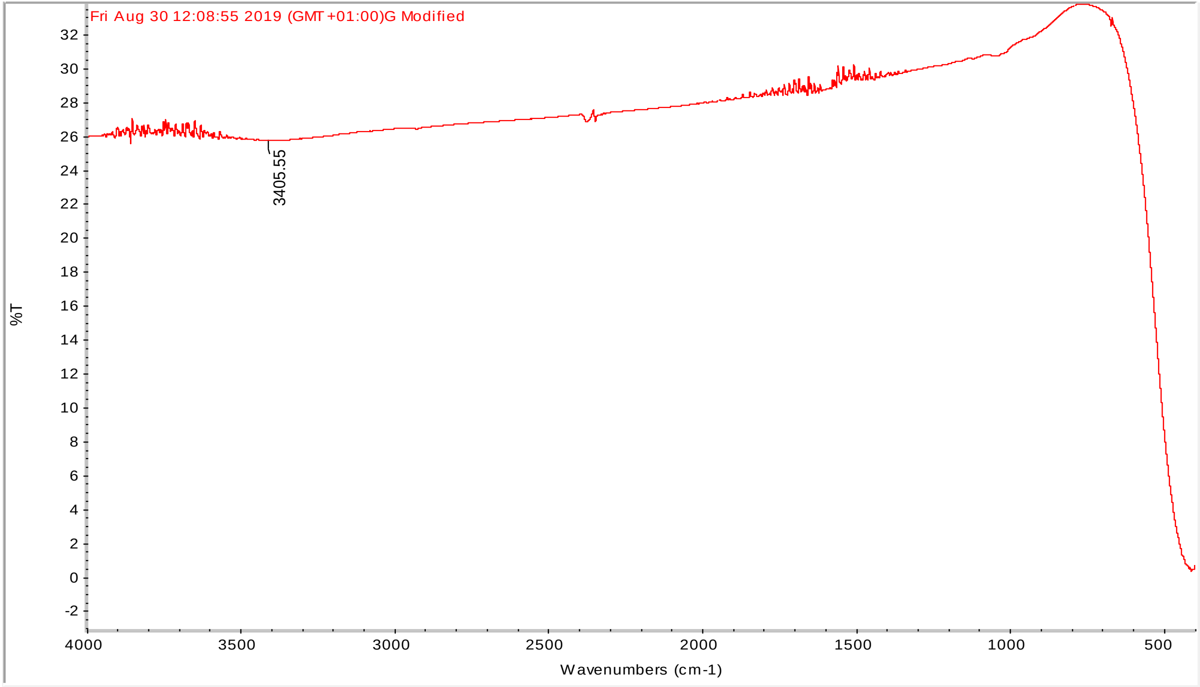

Fig. 1: FTIR spectrum of graphite

Another broadband was found at 1652 cm^{-1} and is assigned to C-O stretch (Si and Samuski, 2008). It is interesting to note that whereas the FTIR of normal graphene oxide has several peaks (Fig. 3) assigned to OH bond (3240 cm^{-1}), carboxyl group (1720 cm^{-1}) (Krasteva *et al*., 2021), stretching and bonding vibration of OH (1600 cm^{-1}) and C-O vibration at 1060 cm^{-1}. (Bali *et* al., 2021). The FTIR of the synthesised nano-graphene oxide shows only two prominent peaks. According to Si and Samulski (2008), such spectra indicate the presence of oxygenated functional groups in the nanographene oxide. Similar findings have been reported by Bera *et al*. (2018), Muthu *et al*. (2022) and Nebl'sin *et al*. (2020).

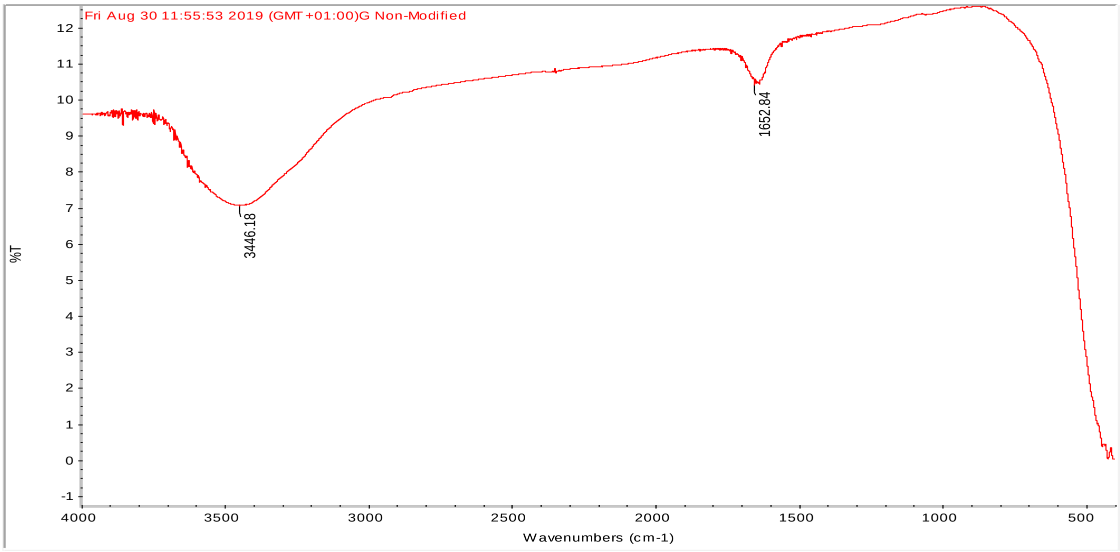

Fig. 2: FTIR spectrum of nanographene oxide

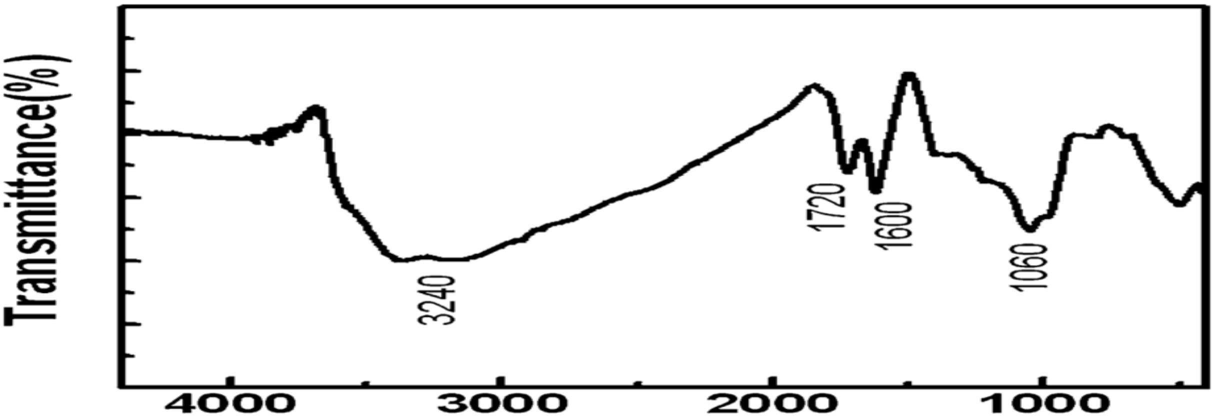

Fig. 4.3: FTIR spectra of graphene oxide
(Source: Ji, F., Li, Y., Feng, J., Su, D., Wen, Y., Feng, Y. and Hou, F. (2009). Electrochemical performance of graphene nanosheets and ceramic composites as anode for lithium batteries. Journal of Materials Chemistry 19: 9063–9067)

The surface morphology of graphite and GO were studied using a scanning electron microscope as shown in Fig. 4.

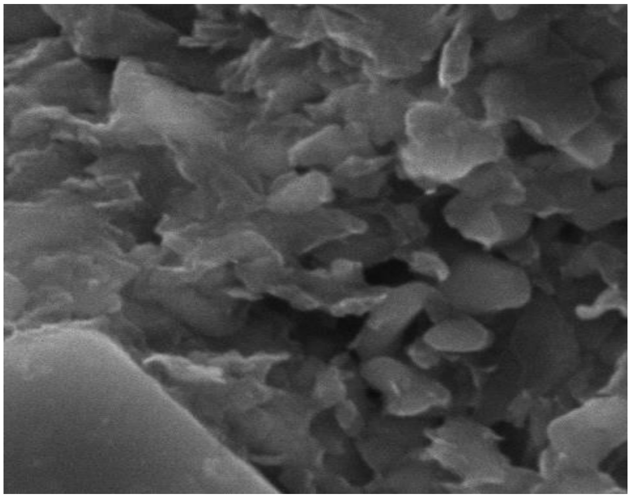

Fig. 4: SEM diagram of nanographene oxide

The micrograph reveals the stacked layer structure of graphite. The figure further shows evidence that the GO consists of randomly aggregated crumpled nanosheets closely associated with each other by strong interaction and the morphology is distinctly different from that of graphite (Fig. 5). The crumpling was due to exfoliation and restacking of GO sheet.

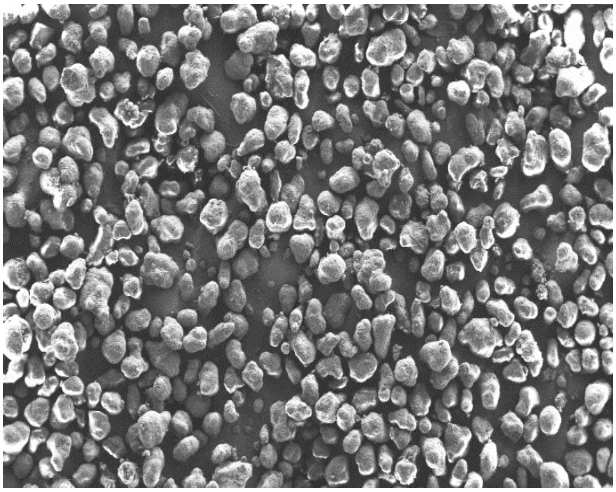

Fig. 5: Scanning electron micrograph of graphite dust

4.0 Conclusion

The present study adds to the literature claims that nanographene dust can be produced from graphite dust using Hummer's method. The yield of the product was 87% and the product shows distinguished features from the precursor (i.e. graphite duest) considering their FTIR spectra and scanning electron micrographs. The FTIR spectra did not show crowded functional groups and compared favourably with the typical spectrum observed for nanographene oxide.

5.0 References

Affraid, R. J. (2023). Large-scale production and application of graphene oxide nanoparticles to meet agriculture needs. Current Applied Science and Technology, https://doi.org/10.55003/cast.2023.254564.

Alzubaidy , F. H. ., Al-Hadad , A., & Abdul-Zahra , N. I. (2023). Biosynthesis of Reduced graphene oxide nanoparticles from Uropathogenic K.oxytoca. *Medical Science Journal for Advance Research*, 4, 1, pp. 47–52. https://doi.org/10.46966/msjar.v4i1.108.

Bali, D. E., Arafa,M. F., Gamaleldin, N. M. & El Maghraby, G. M. (2021). Nanographene oxide for enhanced dissolution rate and antibacterial activity of cefdinir. *Journal of Drug Delivery Science and Technology*,62, https://doi.org/10.1016/j.jddst.2021.102411.

Baragaño, D., Forján, R., Welte, L. *et al.* (2020). Nanoremediation of As and metals polluted soils by means of graphene oxide nanoparticles. *Sci Rep.*, *10*, 1896, https://doi.org/10.1038/s41598-020-58852-4.

Bera, M., Yadav, C., Gupta, P. and Maji, P. K. (2018). Facile One-Pot Synthesis of Graphene Oxide by Sonication Assisted Mechanochemical Approach and Its Surface Chemistry. Journal of Nanoscience and Naotechnology 18(2): 902-912.

Bolibok, P., Szymczak, B., Roszek, K., Terzyk, A.P. & Wiśniewski, M. A. (2021). New Approach to Obtaining Nano-Sized Graphene Oxide for Biomedical Applications. *Materials* , *14*, 1327. https://doi.org/10.3390/ma14061327.

Din, I., Khan, I., Aziz, F., Sher, A. & Hakim, S. (2023). Graphene oxide nanoparticles a remedy for chromium pollution, Current Research in Green and Sustainable Chemistry, 7, https://doi.org/10.1016/j.crgsc.2023.100372.

Eddy, N. O., Garg, R., Garg, R., Aikoye, A. & Ita, B. I. (2022b). Waste to resource recovery: mesoporous adsorbent from orange peel for the removal of trypan blue dye from aqueous solution. *Biomass Conversion and Biorefinery*, DOI: 10.1007/s13399-022-02571-5

Eddy, N. O., Garg, R., Garg, R., Eze, S. I., Ogoko, E. C., Kelle, H. I., Ukpe, R. A., Ogbodo, R. & Chijoke, F. (2023a). Sol-gel synthesis, computational chemistry, and applications of Cao nanoparticles for the remediation of methyl orange contaminated water. Advances in Nano Research, https://doi.org/10.12989/anr.2023.15.1.000.

Eddy, N. O., Odiongenyi, A. O., Garg, R., Ukpe, R. A., Garg, R., El Nemir, A., Ngwu, C. M. % Okop, I. J. (2023b). Quantum and experimental investigation of the application of *Crassostrea gasar* (mangrove oyster) shell–based CaO nanoparticles as adsorbent and photocatalyst for the removal of procaine penicillin from aqueous solution. *Environmental Science and Pollution Research*, doi:10.1007/s11356-023-26868-8.

Eddy, N. O., Ukpe, R. A., Ameh, P., Ogbodo, R., Garg, R. & Garg, R. (2022a). Theoretical and experimental studies on photocatalytic removal of methylene blue (MetB) from aqueous solution using oyster shell synthesized CaO nanoparticles (CaONP-O). Environmental Science and Pollution Research, https://doi.org/10.1007/s11356-022-22747-w

Ji, F., Li, Y., Feng, J., Su, D., Wen, Y., Feng, Y. & Hou, F. (2009). Electrochemical performance of graphene nanosheets and ceramic composites as anode for lithium batteries. *Journal of materials chemistry*, 19, pp. 9063–9067

Karteri, I., Karatas, S., Al-Ghamdi, A. & Yakuphanoglu, F. (2014). The electrical characteristics of thin film transistors with graphene oxide and organic insulators. *Synthetic Metals*, 199, doi. 10.1016/j.synthmet.2014.11.036.

Krasteva, N., Staneva, D., Vasileva, B., Miloshev, G. & Georgieva, M. (2021). Bioactivity of PEGylated Graphene Oxide Nanoparticles Combined with Near-Infrared Laser Irradiation Studied in Colorectal Carcinoma Cells. *Nanomaterials*, 11, 3061. https://doi.org/10.3390/ nano11113061.

Muthu, M. S., Xavier, S. S. J., Ajith, P. & Anand, D. P. (2022). Preparation and characterization studies of nano graphene oxide. *Materials Today: Proceedings*, 66, 4, pp. 2449-2454,https://doi.org/10.1016/j.matpr.2022.06.367.

Nebl'sin, V. A., Galstyan, V. & Silina, Y. E. (2020). Graphene oxide and its chemical nature: Multi-stage interactions between the oxygen and graphene. *Surfaces and Interfaces*, 21, https://doi.org/10.1016/j.surfin.2020.100763.

Odoemelam, S. A., Oji, E. O., Eddy, N. O., Garg, R., Garg, R., Islam, S., Khan, M. A., Khan, N. A. & Zahmatkesh, S. (2023). Zinc oxide nanoparticles adsorb emerging pollutants (glyphosate pesticide) from aqueous solution. *Environmental Monitoring and Assessment*, **https://doi.org/10.1007/s10661-023-11255-0.**

Ogoko, E. C., Kelle, H. I., Akintola, O. & Eddy, N. O**.** (2023). Experimental and theoretical investigation of *Crassostrea* gigas (gigas) shells based CaO nanoparticles as a photocatalyst for the degradation of bromocresol green dye (BCGD) in an aqueous solution. *Biomass Conversion and Biorefinery*.

https://doi.org/10.1007/s13399-023-03742-8

Oukhrib, M., Tamegart, L., Assafi, A., Hejji, L., Azzouz, A., Villarejo, L. P., Haida, M., Mohamed, C. & Gamrani, H. (2023). Effects of graphene oxide nanoparticles administration against reserpine-induced neurobehavioral damage and oxidative stress in an animal model of Parkinson's disease. *Food and Chemical Toxicology*, 178, https://doi.org/10.1016/j.fct.2023.113904.

Rhazouani, A., Gamrani, H., El Achaby, M/. Aziz, K., Gebrati, L., Uddin, M. S. & Aziz, F. (2021). Synthesis and Toxicity of Graphene Oxide Nanoparticles: A Literature Review of *In Vitro* and *In Vivo* Studies. Biomed Res Int., 5518999. doi: 10.1155/2021/5518999.

Si, Y. & Samuski, E. (2008). Synthesis of water soluble graphene. Nano Letters 8, 1679, https://doi.org/10.1021/nl080604h.

Singh, S., Hasan, M. R., Sharma, P. & Narang, J. (2022). Graphene nanomaterials: The wondering material from synthesis to applications. Sensors International, 3, https://doi.org/10.1016/j.sintl.2022.100190.

Tas, A. & Cakmak, N. K. (2020). Synthesis of PEGylated nanographene oxide as a nanocarrier for docetaxel drugs and anticancer activity on prostate cancer cell lines. *Human and Experimental Toxicology*, https://doi.org/10.1177/096032712095.

Uzhviyuk, S. V., Khramtsov, P. V., Raev, M. B. *et al.* (2023). Interaction of graphene oxide nanoparticles with human mononuclear cells in the cell-iq system. *Bull Exp Biol Med*. 175, pp. 172–178. https://doi.org/10.1007/s10517-023-05830-1.

Wei, Q., Pei, S., Wen, G., Huang, K., Wu, Z., Liu, Z., Ma, W., Cheng, H. & Rwn, W. (2019). High yield controlled synthesis of nano-graphene oxide by water electrolytic oxidation of glassy carbon for metal-free catalysis. *ACS Nano*, *13* (8), 9482-9490, DOI: 10.1021/acsnano.9b04447

Yu, H., Zhang, B., Bulin, C. *et al.* (2016). High-efficient Synthesis of Graphene Oxide Based on Improved Hummers Method. *Sci Rep* **6**, 36143, https://doi.org/10.1038/srep36143.

Data availability

All data used in this study will be readily available to the public.

Consent for publication

Not Applicable

Availability of data and materials

The publisher has the right to make the data Public.

Competing interests

The authors declared no conflict of interest.

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Authors' contributions

All the authors participated in the bench work and in the development of the manuscript.