© DEC 2025 | IRE Journals | Volume 9 Issue 6 | ISSN: 2456-8880
DOI: https://doi.org/10.64388/IREV9I6-1712516

Exploration of Liquid-Phase Exfoliation for Graphene Production from Bulk Graphite

ABDUL DAHIRU ARDO BUBA[1], TAOFEEQ AKOREDE ATANDA[2], MEDINA UMAR-OSUMA[3], RAMALAN ABUBAKAR[4]
[1,2,3,4]*Department of Physics, Faculty of Science, University of Abuja, Abuja, Nigeria*

Abstract- ***The demand for sustainable material in technological industries have sparked the discovery of a wonderful material known as GRAPHENE, including its efficiency in its application in various sectors because of its unique properties ranging from electrical, mechanical and thermal properties. However, the conventional methods has cause the researchers in detecting its chemical structure and its temperature which are hazardous to human health and environment. This research deals with eco-friendly method of synthesis using Liquid-phase exfoliation which emerged as a prominent approach for graphene production due its scalability, cost-effectiveness and environmental friendly characteristics. This method of synthesis is employed in order to optimize liquid-phase exfoliation using acetone and deionized water. The scanning Electron Microscope analysed at a magnification of 5µm which reveals crumpled and layered-graphene sheets, which is due to the high flexibility of graphene sheets, which tend to fold, wrinkle and overlap during the exfoliation and drying process while Raman spectroscopy unveiled the sample displayed with three characteristics features called the D-band which is visible at approximately 1350cm^{-1} where the peak indicates the presence of defects within the graphene structure, and the G-band centered around 1580cm^{-1} corresponding to sp^{2}hybridized carbon atoms in the graphene sheet, its strong intensity confirms the presence of graphitic domains and highlighted the graphitic structure of the exfoliated graphene, and the 2D-band produced at 2700cm^{-1} was observed and indicates the few-layer nature of the graphene which are sharp and symmetric while X-Ray Diffraction data confirms successful exfoliated graphite into few-layer graphene from the peak at 22.1° (002 plane) with broadening reflects reduced stacking order and increased interlayer spacing where the absence of sharp peaks indicates the material transitional from crystalline graphite to disorder, few-layer graphene***

Keywords: Exfoliation, Acetone, Deionized water, scanning Electron Microscope, Raman Spectroscopy, X-ray diffraction, D-band, G-band and 2D-band

I. INTRODUCTION

Graphene, a groundbreaking two-dimensional material, exhibits exceptional electrical, thermal, and mechanical properties, positioning it as a cornerstone for next-generation technologies. Novoselov and Geim were awarded 2010 Nobel Prize in Physics for the wonderful research on the graphene.

Its vast potential covers round various industries, including electronics, biomedical uses and energy storage applications, fueling an increase worldwide surge in demand. Liquid phase exfoliation is one of the several synthesis techniques, which becomes a popular method for producing graphene because of its scalability, cost-affordability, and environmental friendliness which was demonstrated by Hernandez in 2008. In contrast to chemical vapor deposition (CVD) or epitaxial growth, Liquid Phase Exfoliation using mechanical methods and solvents to exfoliate graphite into graphene layers. This eliminates the need for high temperatures and expensive equipment which associates with these processes.

Despite its benefits, the Liquid-Phase Exfoliation process has a number of challenges, including low yield, uneven or inconsistent quality, and the environmental effects of the organic solvents that are frequently used in the procedure.

Although liquid phase exfoliation provides a feasible method for producing graphene, questions about its effectiveness and environmental impact still needs more concentration. It`s industrial application is limited by the emphasis on hazardous organic solvents combined with low exfoliation efficiency. Achieving industrial-scale graphene manufacturing requires addressing these problems by improving process parameters and finding sustainable substitutes.

In order to increase yield and quality while reducing environmental damage, recent advancement in research concentrates on optimizing Liquid-Phase Exfoliation parameters, such as solvent selection, sonication duration, and centrifugation speeds, as well as investigating green solvents, such as water-

based or bio-derived alternatives. The goal of these initiatives is to make liquid phase exfoliation a more dependable and sustainable process for creating high-quality graphene, guaranteeing its use in environmentally aware industrial and scientific applications.

Furthermore, there is need to deal with the bonding of graphite and graphene which is more important in order to achieve the high-quality graphene separated from the graphite. Graphene is made up of 2p-orbitals and 1s-orbital through sp^2 hybridization. The structure becomes trigonal planar due to its sp^2 which results in a sigma (σ) connection between the two carbon atoms. Compared to cubic diamond, the graphene structure has a shorter carbon atom-to-atom spacing of 1.42Å. Additionally, graphene has π bonds in its p orbital states that are created when two carbon atoms establish a covalent bond. The л bond shows that the graphene layer has clouds of electrons above and below which is responsible for its semi-metallic nature.

Two main types of graphene synthesis can be distinguished. both the top-down and bottom-up methods. Mechanical exfoliation of graphite and chemical exfoliation of graphene oxide are the two commonly used top-down methods. On the other hand, chemical vapor deposition and epitaxial thermal growth of graphene on substrate are the most often used techniques for bottom-up approaches.

The goal of the top-down method is to separate graphite, the precursor to graphene, into atomic layers. A stack of monoatomic graphene layers joined by weak van der Waals interactions is called graphite. These graphite layers must be separated by overcoming the weak van der Waals pressures which was achieved through ultrasonication.

This practical was tested by Güler in 2021 which happens to be a crucial factor in the process of choosing the solvent used for exfoliation, a low-energy ball mill was used to micromechanically exfoliate hexagonal graphite powders. The solvent utilized to produce graphene were N-Methul-2-pyrrolidone (NMP) and N,N-dimethylformamide (DMF). In order to increase production efficiency, sodium dodecyl sulphate (SDS) and naphthalene were added to the solvents. By examining the graphene, all liquid media yielded 3-5 layers of graphene, however the quantity of high quality of the graphene produced varied between samples. The exfoliation technique yielded a 25% production efficiency in the sample while Li Z in 2020 investigated liquid phase exfoliation as the primary technique for producing two-dimensional (2D) materials, including graphene, in large quantities with a good balance between quality and cost which was widely used by both the academic and industrial sectors. The force produced by ultrasound and the interaction with the solvent molecules have typically been the only explanations for the fragmentation and exfoliation process. However, little to nothing is understood about how they actually happen, such as how big, thick graphite crystals can be exfoliated into tiny, thin graphene flakes. Here, we show that the transition from graphite flakes to graphene occurs in three different stages during ultrasonic liquid phase exfoliation. Large flakes are first ruptured by sonication, and the surfaces of the flakes have kink band striations, generally in zigzag patterns. Second, these striations develop fissures that, when combined with solvent intercalation, cause thin graphite strips to unzip and peel off, eventually exfoliating into graphene. The results will be crucial in the effort to maximize the yield, thickness, and lateral dimensions of graphene and other 2D materials in large-scale liquid phase exfoliation for a variety of uses. Green manufacturing, sometimes referred to as sustainable or environmentally conscious manufacturing, focusses on creating materials in ways that minimize waste, energy use, and hazardous emissions which basically the mission of the thesis.

II. MATERIALS AND METHODOLOGY

Preparation of Solvent Mixture

I prepared a 4:1 (v/v) solvent mixture of acetone and de-ionized water. The ratio was optimized to strike a balance between environmental concerns and exfoliation efficiency.

Graphite Dispersion

In a clean beaker, I Weighed out 1 g of graphite powder and added it to 100 mL of the acetone-water solvent mixture. The mixture was stirred with a magnetic stirrer for 30 minutes to guarantee that the graphite was evenly distributed throughout the solvent, where a magnetic stirrer was used to agitate the mixture for half an hour.

Exfoliation via Ultrasonication

I transferred the dispersion into a glass container with a flat bottom where I later submerged it in water in

© DEC 2025 | IRE Journals | Volume 9 Issue 6 | ISSN: 2456-8880
DOI: https://doi.org/10.64388/IREV9I6-1712516

an ultrasonic bath until the halfway point was reached. In order to exfoliate the graphite into layers of graphene, I then sonicated the mixture for 6hours at 40 kHz, and to avoid overheating, ice should be added to the ultrasonic bath on a regular basis to maintain the temperature at ≤30°C.

Centrifugation

I transferred the dispersion to centrifuge tubes after sonication. To separate the exfoliated graphene from unexfoliated graphite and other particles, I centrifuged the mixture for 30 minutes at 5,000 rpm. I then carefully transferred the exfoliated graphene-containing supernatant into a sanitized container.

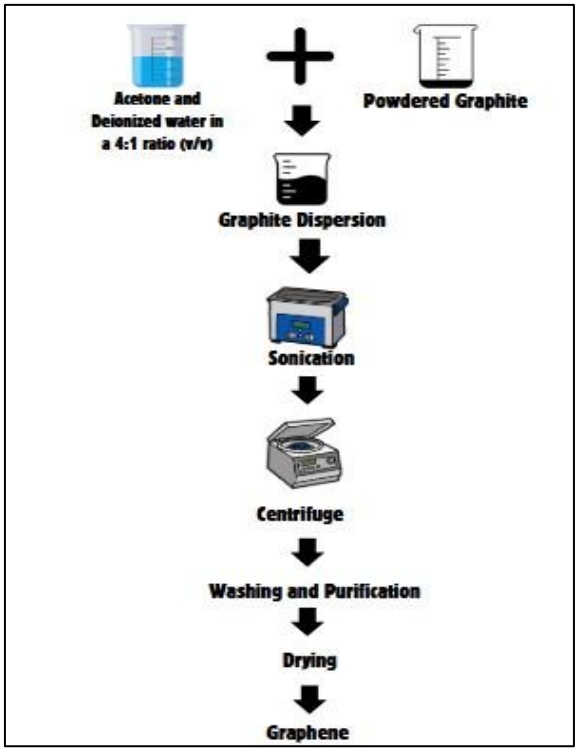
Washing and Purification

I used a vacuum filtration apparatus to filter the exfoliated graphene suspension via 0.2 µm membrane filter. I used 100ml of de-ionized water in order to get rid of any remaining acetone and contaminants to wash the gathered graphene flakes on the filter.

Drying

After washing the graphene flakes, I put it in a glass petri dish. I Dried it for 12hours at 60°C in a vacuum oven to remove any remaining water and solvents.

The diagram below shows the set up of the graphene production from graphite.



Schematic image of the synthesis process

III. RESULT AND DISCUSSIONS

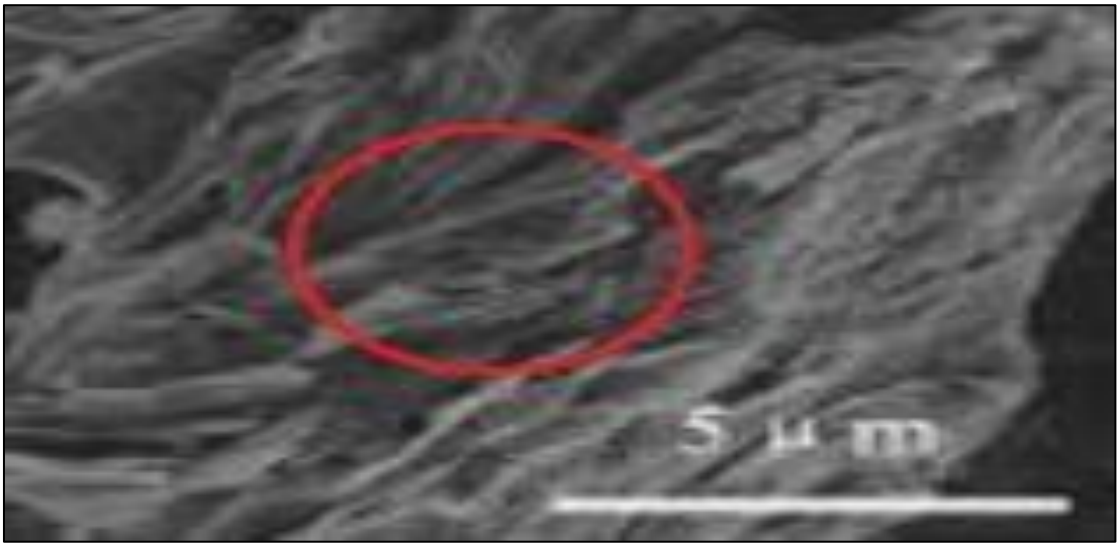


Figure 1: SEM image of graphene derived from Liquid-Phase Exfoliation

The SEM image shows a magnification of 5µm which reveals crumpled and layered graphene sheets, which is due to the high flexibility of graphene sheets that tends to fold, wrinkle, and overlap during the exfoliation and drying process. With the red-circled region emphasizing thinner layers with overlapping or wrinkled features. This is characteristic of graphene synthesized highlighted a successful delamination of graphite into thinner graphene layers.

© DEC 2025 | IRE Journals | Volume 9 Issue 6 | ISSN: 2456-8880
DOI: https://doi.org/10.64388/IREV9I6-1712516

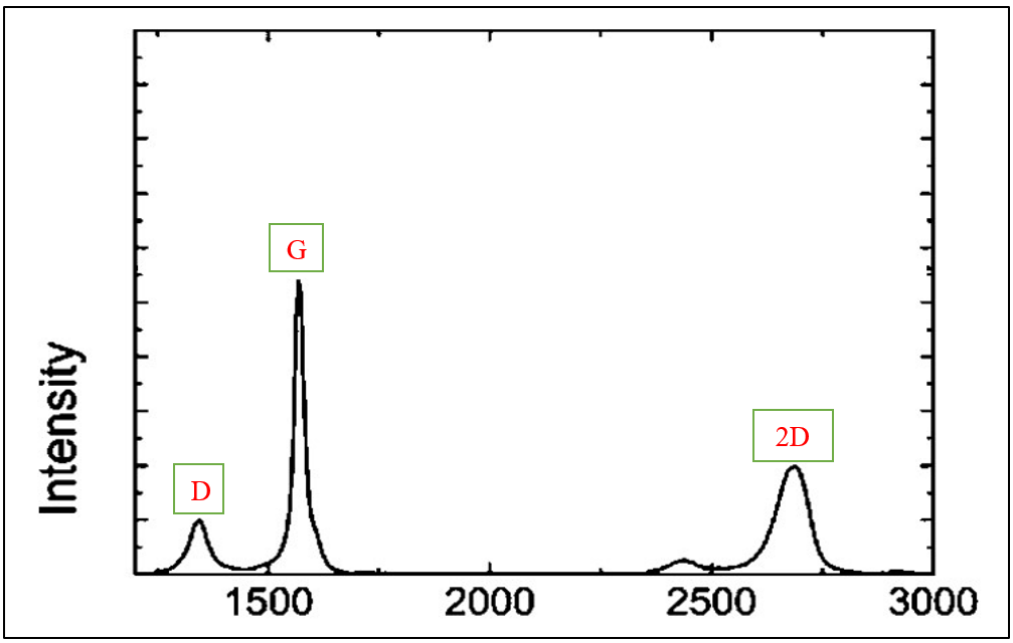


Figure 2: Raman spectrum for graphene derived from LPE

The figure above displayed the sample with three characteristic features for exfoliated graphene, known as the D band, G band, and 2D band. The D-band is visible at approximately 1350 cm^{-1}. This peak indicates the presence of defects or disorder within the graphene structure. The G-band, centered on 1580 cm^{-1} which corresponds to sp^2-hybridized carbon atoms in the graphene sheet. Its strong intensity confirms the presence of graphitic domains and highlights the graphitic structure of the exfoliated graphene. The 2D-band (at ~2700 cm^{-1}) is observed and indicates the few-layer nature of the graphene. In pristine single-layer graphene, the 2D-band is sharp and symmetric. However, here it is broader and less intense, consistent with few-layer graphene.

The figure 3 below is the X-Ray Diffraction data that confirms successful exfoliation of graphite into few-layer graphene. The peak at 22.1° (002 plane) with broadening reflects reduced stacking order and increased interlayer spacing. The absence of sharp peaks indicates the material has transitioned from crystalline graphite to disordered, few-layer graphene.

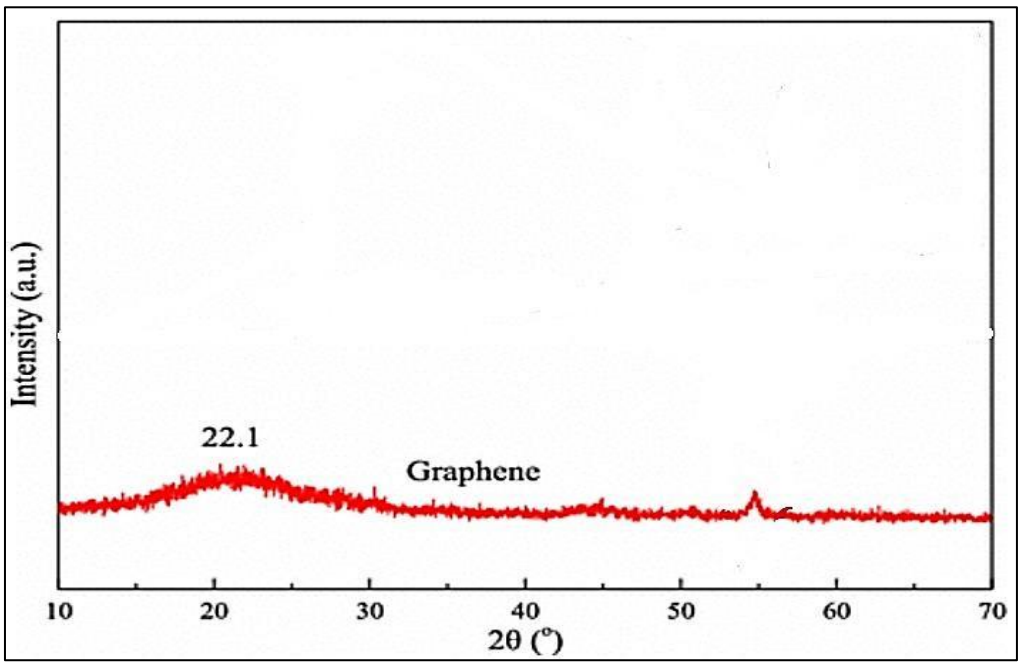


Figure 3: XRD for graphene derived from LPE

IV. CONCLUSION

In summary, the thesis successfully synthesized graphene by optimizing liquid-phase exfoliation of graphite using acetone and de-ionized water as solvents; the results obtained from characterization techniques confirmed the presence of graphene with a moderate level of structural integrity and defects; the Scanning Electron Microscopy image revealed thin, wrinkled sheets typical of exfoliated graphene; the red-marked region highlighted the presence of layered structures with a high degree of exfoliation, consistent with the breakdown of bulk graphite. Three distinctive characteristics of exfoliated graphene were shown by the Raman Spectroscopy results: the existence of flaws or disorder in the graphene structure, such as edge defects and vacancies created during the exfoliation process, was revealed by the D-band at 1350 cm^{-1}. The graphene`s graphitic, sp^2-hybridized carbon structure was confirmed by the G-band at 1580 cm^{-1}, while the few-layer nature of the graphene is indicated by the 2D-band at about 2700 cm^{-1}. The successful exfoliation of graphite into few-layer graphene was confirmed by the XRD patterns, which revealed a broad peak at 22.1° (2θ), which corresponds to the (002) plane of graphene. The peak shifted from 26.5° (graphite) to 22.1°, indicating an increase in interlayer spacing. Overall, the results revealed that few-layer graphene with a balance of structural integrity and moderate flaws was successfully created by liquid-phase exfoliation employing acetone and de-ionized water as solvents. These discoveries provide a scalable and economical way to synthesize graphene, which has potential for other uses in nanotechnology and advanced materials.

V. RECOMMENDATION

Based on the progress achieved in synthesizing graphene through liquid-phase exfoliation (LPE) of graphite using acetone and de-ionized water as solvents, further efforts should focus on enhancing the efficiency and scalability of the process. Future research could explore the use of alternative eco-friendly solvents and solvent combinations to improve exfoliation efficiency while reducing environmental impact. Additionally, optimizing parameters such as sonication duration, energy input, and exfoliation temperature will be critical to maximizing graphene yield and minimizing defects. Collaboration with industries and interdisciplinary researchers in materials processing, chemistry, and environmental engineering will be beneficial in advancing the LPE method towards practical and commercial applications.

REFERENCES

[1] Abdelkader AM, Cooper AJ, Dryfe RAW, Kinloch IA. Nanoscale 7, 2015, pp. 6944-6956.
[2] Avouris P, Chen Z, Perebeinos V., Nat. Nanotechnol. 2 (2007) 605-15. Advanced Functional materials, peer-reviewed scientific journal, Oct 2018.
[3] Bae. S. H. Kim, Y. Lee, X. Xu, J. Park, Y. Zheng, J. Balakrishnan, T. Lei, Song, Y. (2010) "Roll-to-roll production of 30-inch graphene films for transparent electrodes," Nature nanotechnology, vol. 5,no. 8, pp. 574 - 578.
[4] Balandin, A. A. (2011) "Thermal properties of graphene and nanostructured carbon materials." Nature materials; 10(8):569-581.
[5] Barninghams, J. M. Ruan, F. Edler, A. Tejeda, M. Sicot, A. T. Ibrahimi, Z. Jiang, E. Conrad, C. Berger, C. Tegenkamp, (2013). "Exceptional ballistic transport in epitaxial graphene nanoribbons," arXiv preprint arXiv:1301.5354.
[6] Berger, C.; Song, Z.; Li, X.; Wu, X.; Brown, N.; Naud, C.; Mayou, D.; Li, T.; Hass, J.; Marchenkov, A. N. (2006). Electronic Confinement and Coherence in Patterned Epitaxial Graphene. Science, 312, pp.1191-1196.
[7] Bolotin, K. I.; Sikes, K. J.; Jiang, Z.; Klima, M.; Fudenberg, G.; Hone, J.; Kim, P.; Stormer, H. L. (2008) Ultrahigh Electron Mobility in Suspended Graphene. Solid State Commun, 146, pp. 351-355.
[8] Bunch, J. S; Verbridge, S. S. Alden, J. S. Van Der Zande, A. M. Parpia, J. M. Craighead, H.G and McEuen, P.L (2008) "Impermeable atomic membranes from graphene sheets," Nano letters, vol. 8, no. 8, pp. 2458-2462.
[9] Cai, W., Wang, L., et al. (2010). "Graphene-based materials for solar energy conversion." *Nano Energy*, 1(3), 290-296.
[10] Chapman, R.T, Springate, E, Fromm, F, Raidel, C, Seyller, T, King, P, Parmigiani, F, Grioni, M, Hofmann, P. (2015), Tunable carrier multiplication and cooling in graphene 2015; Nano Lett 15: 326–331.
[11] Chen. J.H, Jang, C. Xiao, S. Ishigami, M and Fuhrer,M.S, (2008). "Intrinsic and extrinsic

performance limits of graphene devices on SiO2," Nature nanotechnology, vol. 3, no. 4, pp. 206-209.

[12] Geim, A. K., & Novoselov, K. S. (2004). The rise of graphene. *Nature Materials, 6*(3), 183191. https://doi.org/10.1038/nmat1849

[13] Geim, A. K., & Novoselov, K. S. (2007). "The rise of graphene." *Nature Materials*, 6(3), 183191.

[14] Geim. A. and Novoselov, K. (2010). "The rise of graphene," Nature materials, vol. 6, no. 3, pp. 183 - 191.

[15] Geim. A.K. Science, 324:1530-1532, 2009.

[16] Hussain, T., Wang, Y., & Yang, M. (2021). Graphene from biomass: A review of synthesis methods and applications. *Materials Today Advances, 10*, 100123. https://doi.org/10.1016/j.mtadv.2020.100123

[17] Zhu, Y., Murali, S., Stoller, M. D., Ganesh, K. J., Cai, W., Ferreira, P. J., & Ruoff, R. S. (2010). Carbon-based materials for energy storage. *Science, 332*(6037), 1537-1541. https://doi.org/10.1126/science.1200770