

Morphological, Elemental Composition and Structural Analysis of Novel Graphite/rGO Hybrid Synthesis via One-Pot Facile Hydrothermal

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How to cite this paper: Allim, M.F., Azam, M.A., Shabdin, M.K., Safie, N.E. and Kosnan, M.A. (2025) Morphological, Elemental Composition and Structural Analysis of Novel Graphite/rGO Hybrid Synthesis via One-Pot Facile Hydrothermal. *Journal of Power and Energy Engineering*, 13, 14-21.
<https://doi.org/10.4236/jpee.2025.1312002>

Received: November 19, 2025

Accepted: December 5, 2025

Published: December 8, 2025

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Abstract

This study reports a facile and novel synthesis of a graphite/reduced graphene oxide (graphite/rGO) hybrid material derived entirely from waste palm kernel shell (PKS). To the best of our knowledge, no previous work has successfully hybridized graphite and rGO from a common biomass precursor, nor established a clear analytical route to confirm their hybridization. In this work, PKS-derived graphite and rGO were physically integrated through a facile one-pot hydrothermal method, forming a well-bonded hybrid with enhanced interlayer compatibility. The hybridization was evidenced through energy-dispersive X-ray (EDX) elemental analysis, revealing an optimized carbon-to-oxygen ratio intermediate between that of pure graphite and rGO, confirming the synergistic bonding between sp^2 and partially reduced sp^3 domains. Morphological and structural characterization were also incorporated. This simple approach provides a sustainable and scalable pathway to develop carbon-based hybrid materials for advanced electrochemical applications such as lithium-ion batteries and supercapacitor due to the combined properties from both graphite and rGO attributes.

Keywords

Graphite, Reduced Graphene Oxide, Graphite Hybrid, Hydrothermal, Waste Palm Kernel Shell

1. Introduction

Graphite and graphene-based materials have gained significant attention for energy storage and conversion devices due to their high conductivity, large surface area, and mechanical stability [1]-[3]. Despite extensive research on these carbon allotropes, the combination of graphite and reduced graphene oxide (rGO) into a single hybrid structure has not been realized. Graphite offers excellent electrical conductivity [4], while rGO provides abundant defect sites for ion storage [5] [6]. Also, graphite's interlayer distance is reportedly insufficient for the intercalation of large ions, on the other hand, graphene (as in rGO) stacks can be adjusted by the intercalation of many species, such as polymers, organic molecules, solvent molecules, and functional groups [7]. Hybridizing both materials can potentially bridge the trade-off between conductivity and surface reactivity, as attributes from both precursors are believed to be embedded in the hybridized material to yield a synergistic effect towards various applications such as metal-ion batteries, supercapacitors, photovoltaic, and sensors [6]-[11].

Moreover, the use of biomass-derived carbon precursors offers a sustainable alternative to conventional synthetic routes [1]. Palm kernel shell (PKS), an abundant agricultural byproduct in Malaysia, represents a low-cost, renewable feedstock for high-quality carbon materials [1]-[3]. While PKS has been previously explored for graphite and rGO production, separately, from past literatures [1]-[3] [6] [12], its utilization to generate both graphite and rGO precursors into hybridization has not yet been reported. Notably, a low-cost raw material such PKS is a good alternative in the synthesis of graphene and its derivatives as compared to high-capital technology as noted by [10] on CVD-derived (Chemical Vapor Deposition-derived) graphene is less economical.

In this work, we demonstrate for the first time the facile preparation of a graphite/rGO hybrid derived from PKS. The novelty lies in:

- (1) the utilization of dual carbon source derived from a single biomass precursor,
- (2) the first-ever facile one-pot hydrothermal synthesis hybridization between graphite and rGO, and
- (3) the use of EDX carbon-to-oxygen elemental ratio as a confirmatory technique for hybrid formation.

This study highlights a simple and scalable route for producing sustainable hybrid carbon materials with potential applications in battery and supercapacitor electrodes.

2. Experimental Method

2.1. Synthesis of Graphite/rGO Hybrid

In this study, graphite and rGO are two key raw materials to produce a graphite/rGO hybrid material. Graphite powder that being derived from palm kernel shell waste was supplied by Innovative Ecographe Sdn Bhd as in-kind contribution, whereas rGO was synthesized via a modified Hummer's Method that utilized

greener synthesis route from [6], which using milder reducing agent that is ascorbic acid unlike original method that using sodium borohydride, likewise noted by [5] in which recent modifications towards Hummer's Method is targeted to achieve less risk and non-toxic synthesis.

The hybrid material was synthesized through a facile, one-pot hydrothermal synthesis protocol that produced a 1:1 stoichiometry reactant-product pathway, approximately 100% yield. The procedure kickstarted where 0.95 g of graphite powder and 0.05 g of rGO powder were mixed in a 100mL beaker containing 30 mL of Deionized Water (DI). The mixture was stirred by using magnetic stirrer for 15 minutes, then sonicated for 30 minutes. Subsequently, the mixture was transferred into a 50 mL PTFE-lined hydrothermal autoclave reactor and heated in a vacuum drying oven (180°C, 24 hours). The hydrothermal synthesis reactor was cooled down naturally (no ice bath quenching) to room temperature after the synthesis completed. The hybrid material mixture was poured out from the reactor chamber to a crucible and dried in vacuum oven at 60°C for 24 hours. Later, the dried mixture was well ground into smaller-fine powder by using a mortar and pestle. The graphite/rGO hybrid powder was kept in a dry cabinet prior further characterization [product yield: ~1 g].

2.2. Material Characterization

A series of material characterization approaches were carried out for synthesized pristine graphite, rGO and graphite/rGO hybrid. By using a Field Emission Scanning Electron Microscopy (FESEM), morphological analysis was conducted, [Brand: HITACHI SU4500, operating voltage 10 kV]. Additionally, elemental composition of the materials was analyzed via EDX function through FESEM. To determine structural properties, X-ray Diffraction (XRD) was adopted [Brand: Rigaku Miniflex Benchtop, 2θ (degree) ranges from 0° - 150°, $\lambda = 1.54180$, X-Ray Tube: Copper with scan voltage 40 kV].

3. Results and Discussion

3.1. Morphological Analysis

The morphology of graphite that being derived from natural carbon resources are usually observed as irregular, porous [12] and defined bulky cluster [1] [4] [9], whereas rGO is reported as display of strong aggregation [3] [13] and crumpled [11] [14] wrinkled structure [4] [6] [11] [12] resulting from the efficient elimination of defects and oxygen-based functional groups [13]. Thus, it is believed that combined characteristics of the two materials shall be observed in their hybrid morphology. **Figures 1(a)-(f)** revealed the surface microscopic picture of graphite, rGO and graphite/rGO hybrid studied in this report.

While **Figures 1(a) and 1(c)** correlated to the graphite and rGO characteristics, a well-defined distribution rGO hybridized with graphite observed in **Figure 1(e)**. Indeed, united attributes of the two precursors clearly observed at the hybrid material. This showed the facile one-pot hydrothermal synthesis protocol has suc-

cessfully hybridized graphite and rGO derived from palm kernel shell resources. At the same time, a higher magnification of image for respective materials can be referred from **Figure 1(b)**, **Figure 1(d)**, and **Figure 1(f)**. At magnified view (20,000) compared to the counterpart (5,000), a clearly visual disclosed a better view on graphite bulkiness, rGO's wrinkled sheets and well-hybridized distribution of rGO at hybridized graphite/rGO structures.

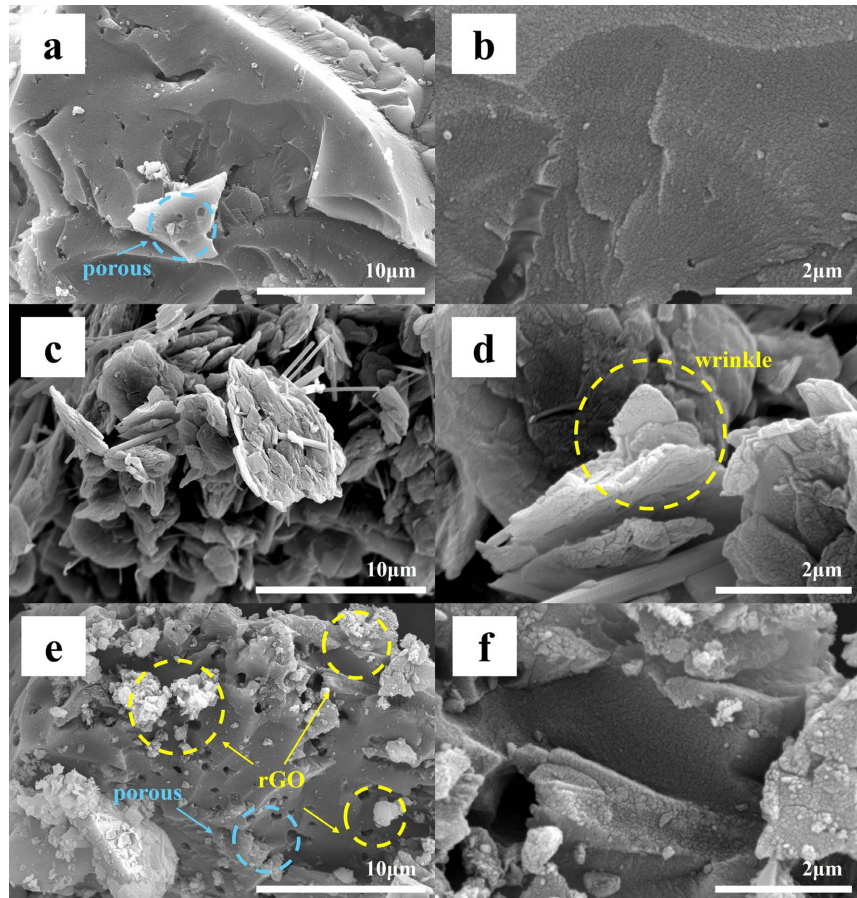


Figure 1. FE-SEM images of graphite at (a) 5000 and (b) 20,000 magnification levels, synthesized rGO at (c) 5000 and (d) 20,000 magnification levels, and graphite/rGO hybrid at (e) 5000 and (f) 20,000 magnification levels.

3.2. Elemental Composition Analysis

The main key indicator from EDX analysis for carbon-related materials is the Carbon-to-Oxygen (C/O) ratio. This is to quantify the extent of oxidation or reduction in the studied carbon materials, serving as a direct indicator of atom modification via chemical or physical, towards its suitable applications.

In this current study, C/O ratio result is used as evaluation tool in proving the hybridization occurrence. As depicted in **Figures 2(a)-(c)**, C/O ratio for graphite, rGO and graphite/rGO hybrid is 15.43, 4.30, and 13.44, respectively. For graphite, it showed that the high carbon content comes from the nature of the PKS raw material itself. Conversely, rGO produced C/O ratio approximately that is pro-

portional to previous findings, ~4 - 5 [6] [15], showing a successful rGO synthesis via modified Hummer's method approach, indicating oxygen functionalities presence through oxidation and further eliminated (reduction) to yield rGO. Contemplating at the C/O ratio, the trend will be graphite > graphite/rGO > rGO. It is believed that the hybrid material possessed a higher oxygen content compared to the graphite due to the contribution of oxygen-containing constituent from the rGO. From **Figure 2(a)**, it is intentionally to conduct EDX analysis for whole full area in order to evaluate the whole surface of graphite, while for rGO and graphite/rGO hybrid, a selected area was chosen to specifically target area of interest such as **Figure 2(b)**, on the wrinkled exfoliated sheets; where insertion of oxygen-containing groups depicted via visual increased inter-planar spacing, showing well-exfoliated rGO [5] and **Figure 2(c)**, at the observed clustered rGO at graphite domain, since graphite bulk whole surface already been analyzed, thus, domain with rGO attachment only was analyzed. This is to further justify the hybridization occurrence between graphite and rGO (95:4 mass basis). Certainly, C/O ratio is a useful tool to quantitate and evaluate the incorporation between graphite and rGO that are both carbon materials.

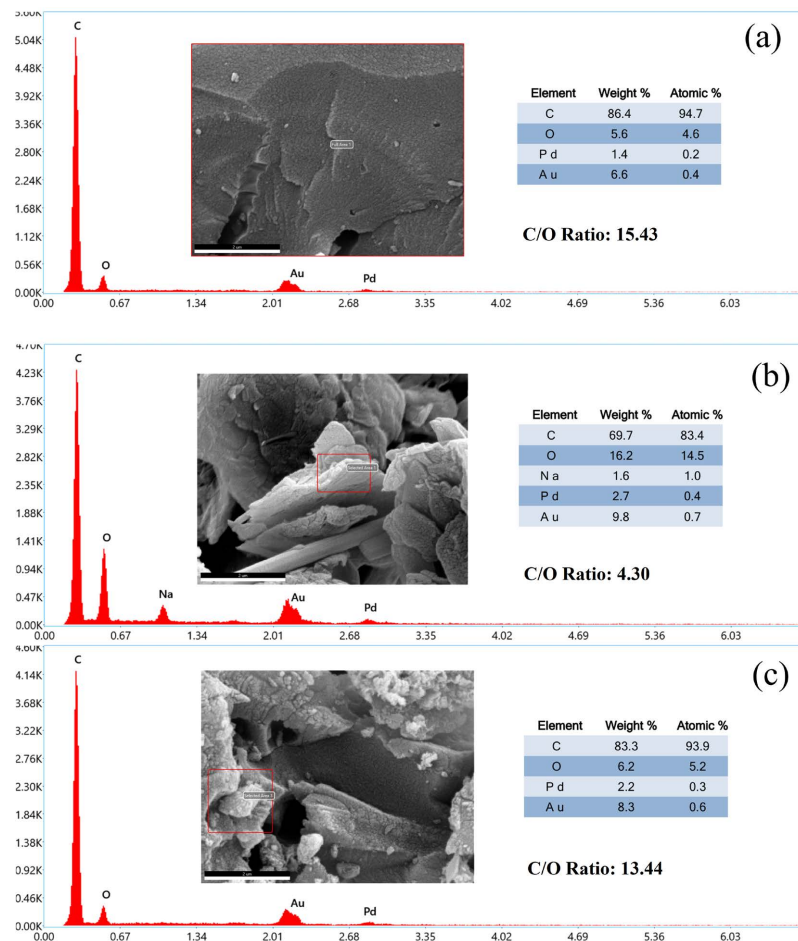


Figure 2. EDX analysis: elemental composition of (a) graphite, (b) rGO, and (c) graphite/rGO hybrid.

3.3. Structural and Crystallinity Analysis

Prominently, XRD diffractogram of graphite-like structures shows peak at about 26.5 and 42.3 correspond to lattice structure [002] and [100], respectively [2]. From **Figure 3**, graphite, rGO, and graphite/rGO hybrid showed [002] lattice peak at 26.93, 26.90, and 26.65 and [100] lattice peak at 43.77, 43.31, and 43.39, respectively. This stacking analysis on these materials structural characterization justified the synthesis procedure in this current study, as referred to past literature [2] [5] [13] [14].

It can be observed that graphite raw material is highly crystalline from the sharp [002] peak due to highly arranged carbon sp³ sp² orbital, whereas a broad peak at the same lattice for rGO showed the defected structure of carbon sp² orbital thanks to the reduction of oxygen-attached functional groups at the structure, disrupting the crystalline graphite as precursor [14]. Concurrently, slightly mixed of sharp-broad peak observed at graphite/rGO hybrid justified that synthesis that taken place that believed due to rGO constituents in the hybrid is approximately 5%, making the lattice to be in a mixed of crystalline and amorphous carbon; contributed from both precursors. An identical phenomenon was observed and noted too, at [100] peak lattice, because the trend observed for [100] lattice is the same for [002] lattice, thus the same interpretation applied.

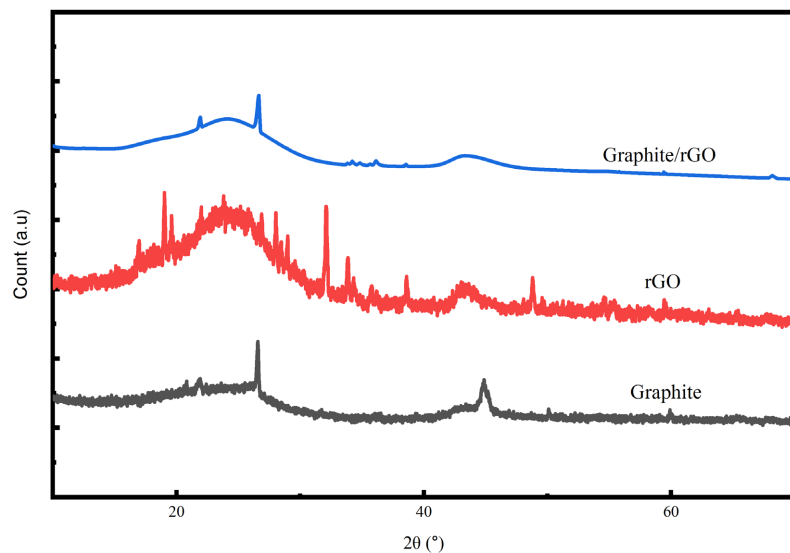


Figure 3. XRD pattern from bottom, (a) graphite, (b) rGO, and (c) graph-ite/rGO hybrid.

4. Conclusion

In conclusion, a facile and novel approach to synthesize a graphite/rGO hybrid entirely from waste palm kernel shell has been demonstrated for the first time. The hybridization was successfully confirmed using EDX elemental analysis based on the C/O ratio, a reliable indicator for interfacial bonding in carbon composites. This sustainable and scalable method opens a new avenue for developing hybrid carbon materials suitable for advanced applications such as lithium-ion battery

and supercapacitor electrodes.

Acknowledgements

The authors gratefully acknowledge Universiti Teknikal Malaysia Melaka (UTeM) for the facility support and appreciate the in-kind contribution of raw material supply which is graphite derived from palm kernel shell given by Innovative Ecographe Sdn Bhd.

Data Availability

Data will be made available upon request.

Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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