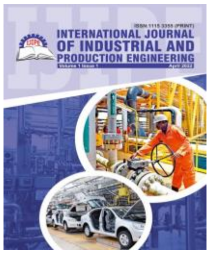

Vol. 3 No. 3 (2025)

INTERNATIONAL JOURNAL OF INDUSTRIAL AND PRODUCTION ENGINEERING (IJIPE)

JOURNAL HOMEPAGE: https://journals.unizik.edu.ng/index.php/ijipe/

Biochemical Synthesis of Zinc-Graphene Nano-Composite Using Cassava Leaf Extract

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ABSTRACT

This research explores eco-friendly methods for creating zinc–graphene nanocomposites through green synthesis techniques using plant materials. The study used cassava (Manihot esculenta) leaf extract as a natural substance to reduce and stabilise the nanocomposite. Energy Dispersive X-ray (EDX) spectroscopy were used to identify the elements and stability of the produced material. The EDX analysis demonstrated that the primary elements of the nanocomposite were zinc and carbon from graphene oxide, which measured 44.50% and 20.70%, respectively. The 15.00% oxygen measurement confirmed the existence of graphene oxide within the material. The analysis identified trace elements, including magnesium (3.62%), calcium (7.01%), iron (3.60%), sulphur (2.40%), sodium (2.02%), and potassium (1.15%), which were likely derived from cassava extract or reaction intermediates. These components have the potential to introduce extra catalytic properties as well as biological functions to the material. The research findings confirm the feasibility of sustainable bio-based methods for creating advanced nanomaterials. The research highlights material science's progressive movement towards environmentally sustainable nanotechnology approaches.

Keywords: Green Synthesis; Cassava Leaf Extract; Nanotechnology; Zinc-Graphene Oxide; Nanocomposite.

1. Introduction

Nanotechnology has largely revolutionized materials science by modifying chemical, physical and mechanical properties of various materials as numerous studies indicate (Rafique et al., 2020; Malik et al., 2023; Kumar et al., 2024; Das et al., 2025). The carbon-based nanocomposites and metal oxide that integrate zinc oxide (ZnO) and graphene oxide (GO) are outstanding compared to other nanomaterials because of the synergistic effects (Hassan et al., 2021; Rehman et al., 2022; Sharma et al., 2024). ZnO/GO nanocomposites have great optical electrical mechanical and catalytic performances which qualify them to be used in sensing applications photocatalysis drug delivery systems and corrosion protection (Yaqoob et al., 2020; Kachere et al., 2021; Albiter et al., 2022; Arlarasu et al., 2022; Ebrahimi et al., 2023).Conventional chemical vapour deposition, sol-gel processing and hydrothermal techniques require toxic chemicals and operate at high temperatures and extreme energy consumption (Boukhoubza et al., 2020; Ahmed et al., 2023; Sugianto et al., 2023).

The disadvantages of traditional synthesis methods have led researchers to develop greener and sustainable approaches to synthesis, which now include plant-based green synthesis methods as a viable option. Phytochemicals like flavonoids, phenols, tannins, polyphenols and polysaccharides found in plant extracts serve as natural reducing and stabilising agents throughout nanoparticle formation processes (Shafey, 2020; Soni et al., 2021; Adeyemi et al., 2022). The cassava leaf extract from Manihot esculenta, which contains numerous bioactive compounds, has not been fully investigated even though it is available as an agricultural byproduct. This research introduces an innovative method to generate eco-friendly nanomaterials from an underutilised biodegradable resource. The produced composite provides strong anti-corrosion defence in industrial settings alongside compliance with global sustainability and waste valorisation objectives. This research advances eco-conscious nanotechnology through the combination of waste reduction techniques with high-level material development practices.

2. Materials and Methods

2.1 Materials

The key materials used for this study comprised graphene flakes combined with zinc acetate, along with Manihot esculenta (cassava) leaf extract and different solutions, including hydrochloric acid (HCl), sulphuric acid (H_2SO_4), sodium nitrate ($NaNO_3$), hydrogen peroxide (H_2O_2), potassium hydroxide (KOH), acetone, ethanol, distilled water, and aluminium foil.

2.2 Method

2.2.1 Preparation of the Manihot esculenta leaf extracts (Cassava)

Fresh cassava leaves (Manihot *esculenta) were* collected within the premises of the Federal University of Petroleum Resources, Effurun, Delta State, Nigeria. The fresh leaves were washed with water and then allowed to dry for two weeks at room temperature. The leaves were ground into fine powder for subsequent generation of plant extract. The mixed solution started cooling naturally until reaching room temperature, after which filtration produced a clear extract. The bioreducing and stabilising agent for nanoparticle synthesis was prepared using the filtrate obtained from the extract.

Figure 1: pulverized Cassava leaves

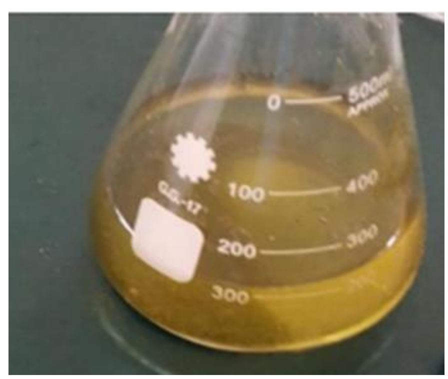

Figure 2: Sample of prepared plant extract

2.2.2 Biosynthesis of Zinc Nanoparticle

Synthesis of zinc oxide nanoparticles occurred through a green approach which modified the Hummers' method. The synthesis combined 40 mL of cassava leaf extract obtained from Manihot esculenta with 500 mL of zinc acetate solution at a concentration of 0.01 M. A magnetic stirrer delivered dropwise NaOH solution into the mixture, which received continuous stirring. The reaction solution remained at 70°C for about four hours and displayed a yellow colour transformation into whitish cream due to zinc acetate reduction into zinc hydroxide [$Zn(OH)_2$] precipitate formation. The reaction produced a precipitate that received drying procedures to obtain zinc oxide nanoparticles. Figures 3 and 4 depict the synthesis process and zinc oxide nanoparticles.

Figure 3: Synthesis of zinc Nano-particles

Figure 4: Zinc-oxide Nano-particles

2.2.3 Synthesis of Graphene Nanoparticle

The modified Hummers' method was used to create graphene oxide (GO). 4.0 g of graphite flakes and 2.0 g of sodium nitrate ($NaNO_3$) were mixed together with 200 mL of concentrated sulphuric acid (98% H_2SO_4) in a beaker. An ice bath kept the beaker covered with aluminium foil at 0°C to prevent exothermic reactions. The mixture was subjected to stirring with a magnetic stirrer at room temperature for four hours once it cooled down. The experimental procedure continued with the addition of 12.0 g of potassium permanganate ($KMnO_4$) to the mixture in a gradual manner while stirring continuously to prevent thermal runaway or violent reactions. The mixture underwent continuous stirring for another 1.5 hours. The reaction mixture received a 100 mL addition of water heated to 80°C followed by 20 minutes of stirring. Afterward, the reaction mixture was slowly added with 80 mL of 30% hydrogen peroxide (H_2O_2). The addition of chemicals caused the solution colour to transform from yellow to dark brown, revealing graphite oxidation. The reaction mixture stayed at 80°C while stirring continued for more 2hrs. As time passed, the solution became black, which demonstrated that graphene oxide was produced. A 160 mL solution of 32% hydrochloric acid (HCl) was used to filter and wash the mixture, which underwent multiple rinses with distilled water until the filtrate achieved a neutral pH (~6). We obtained uniformly sized graphene oxide powder by drying the final product in a laboratory oven followed by grinding and sieving processes shown in Figures 5 and 6.

2.2.4 Zinc-Graphene Nanocompoite

A green synthesis method produced the zinc–graphene nanocomposite through the plant extract-mediated reduction of graphene oxide (GO) to graphene. The nanocomposite was formed by integrating reduced graphene with zinc nanoparticles. In the final mixture, zinc nanoparticles made up 85% of the weight, while graphene accounted for 15%.

Figure 4: Zinc-Graphene Nano-composite after synthesis

2.2.5 Synthesis of (80/15wt.%) Zinc-Graphene Nano-composite

A total of 0.15 g of graphene oxide (GO) was dispersed in 75 mL of cassava leaf extract and stirred with the help of a mechanical magnetic stirrer for 3 hours. This step allowed the subsequent reduction of GO to graphene under the influence of phytochemicals (e.g., flavonoids and polyphenols) contained in the plant extract that can serve as natural reducing agents. This prolonged stirring time resulted in homogeneous reduction and dispersion of GO and increased the surface area that could be used in the attachment of the nanoparticles later. Following the cut-down, a quantity of 0.85 g of the green-synthesised zinc nanoparticles was added to the solution, and the resulting mixture was stirred further at 70°C within 4 hours. This temperature was selected based on the need to facilitate the effective contact between zinc and graphene sheets without affecting the degradation of the bioactive compounds and agglomeration of the nanoparticles.Aluminium foil was then used to cover the mixture in order to reduce photoreduction and contamination and then left to cool to room temperature to slowly solidify and stabilise the composite structure. The finished product was filtered, dried in the oven to get rid of the moisture content and powdered. The nanocomposite sample (about 85 wt. % zinc and 15 wt. % graphene) was characterised by storing it in a dry sample bottle in an airtight manner. The selected composition was premised on the initial optimisation between the electrical and mechanical properties of the reinforcement provided by graphene and the anti-corrosive and photocatalytic activity of zinc oxide. Synthesis conditions, especially the Graphene-to-zinc proportion, reductive atmosphere and temperature of reaction, are crucial to the structural stability, dispersibility and practical activity of the as-obtained Zn/Graphene nanocomposite.

2.2.6 EDX Spectroscopy Characterization

EDX spectroscopy was used to assess the chemical components present in the synthesised zinc-graphene nanocomposites from cassava leaves. The EDX analysis was carried out using a JEOL JSM-7600 loaded with an EDX detector. The samples were placed on a carbon-coated stub and covered with a thin layer of gold to aid the imaging. To conduct EDX testing, electrons were focused on the material's surface to stimulate the atoms and result in images and X-ray signals. After the X-rays were detected, EDX analysed them to determine the composition.

3. Results and Discussion

3.1 Physical Characterization of Synthesized Nanoparticles

The synthesized zinc nanoparticles demonstrated a smooth white crystalline surface morphology which Figure 4 illustrates. The observed characteristics match prior green synthesis research which demonstrated phytochemical reduction methods produced crystalline zinc nanoparticles that were evenly distributed (Iqbal et al., 2021; Kambale et al., 2023). The combination of vivid coloration and crystalline structure indicates successful nucleation and growth processes during synthesis which benefits high-purity surface reactive applications like antimicrobial coatings and corrosion prevention. Figure 5 illustrates that graphene oxide (GO) nanoparticles present as dark black crystalline clusters. Physical evidence supports previous research which shows that plant-extract-derived GO contains oxygen-rich groups that produce its dark appearance and layered structure (Li et al., 2022). The structural crystallinity demonstrates a partially reduced system with preserved oxygen functionalities that result in better dispersibility in water-based solutions critical for sensing and catalytic applications.

3.2 Energy Dispersive X-ray (EDX) analysis

The energy-dispersive X-ray (EDX) spectrum of the graphene oxide (GO) synthesized using cassava leaf extract (Figure 8) revealed the elemental composition as follows: The elemental analysis shows 82.30% carbon (C), 7.47% oxygen (O), 5.21% sulfur (S), 2.60% iron (Fe), and 2.42% silicon (Si). The dominant carbon peak demonstrates the graphene-based material's carbon-rich structure. The elevated amount of carbon present indicates effective graphene oxide reduction which aligns with prior research demonstrating that green synthesis methods usually produce GO materials with carbon content above 80% (Kong et al., 2019; Kothandam et al., 2023). The 7.47% oxygen content in the material indicates graphene surface functionalization with hydroxyl, epoxy, and carboxyl groups. The oxygen-containing groups maintain hydrophilicity and dispersion stability in aqueous solutions which enables later nanocomposite formation. Research shows plant-extract-mediated GO syntheses produce similar oxygen levels which verify phytochemicals' capacity to partially reduce and functionally enrich GO (Kumar et al., 2021; Thiyagarajulu et al., 2024). The cassava leaf extract contained sulfur at 5.21% which originates from sulfur-bearing phytochemicals including thiols and organosulfates. The presence of this element could enhance GO functionality in subsequent nanocomposite applications. The presence of trace elements such as iron (2.60%) and silicon (2.42%) in the sample is believed to stem from the mineral content in cassava leaves. The EDX analysis confirms cassava leaf extract performs well as a green reducing and stabilizing agent. Analysis of the compositional profile demonstrates alignment with established green synthesis techniques and establishes the material's potential for incorporation into zinc-graphene nanocomposites. The composite achieves better performance through metal nanoparticle anchoring because its high carbon and moderate oxygen content creates an optimal balance between structural carbon components and reactive functional groups.

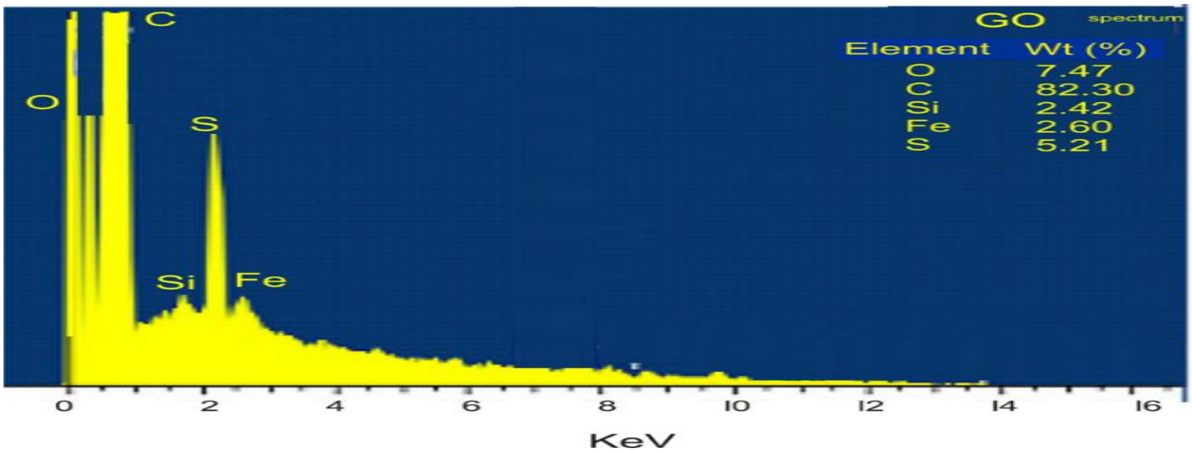

Figure 8: EDX spectrum for synthesized graphene-oxide

The Energy Dispersive X-ray (EDX) spectra depicted in Figure 9 indicates that the zinc-graphene nanocomposite fabricated using a 0.85:0.15 ratio achieved optimal properties. Since zinc makes up 44.50% of the formula, it indicates that the material is successful in incorporating zinc nanoparticles, which are fundamental for increasing its antimicrobial, catalyst and corrosion-resistant qualities. Graphene oxide is present in this composite, due in part to carbon, which provides a big surface area, a tough shape and a conductive feature. Because of its oxygen functions, a portion of oxygen (15%) can be found in graphene oxide and helps bond the zinc with the graphene (Xu et al., 2022). The content of magnesium (3.62%), calcium (7.01%), iron (3.60%), sulphur (2.40%), sodium (2.02%) and potassium (1.15%) might be from residual matter in the cassava leaf extract or caused by reactions during the synthesis process. They may also serve to stabilise the nanocomposite or increase its usefulness in catalysis. The main strength of this material comes from the zinc, and graphene oxide slightly improves its structure and appearance. This sample blends the two elements well, indicating that applying the green route to synthesis can maintain eco-friendly environment and sustainability.

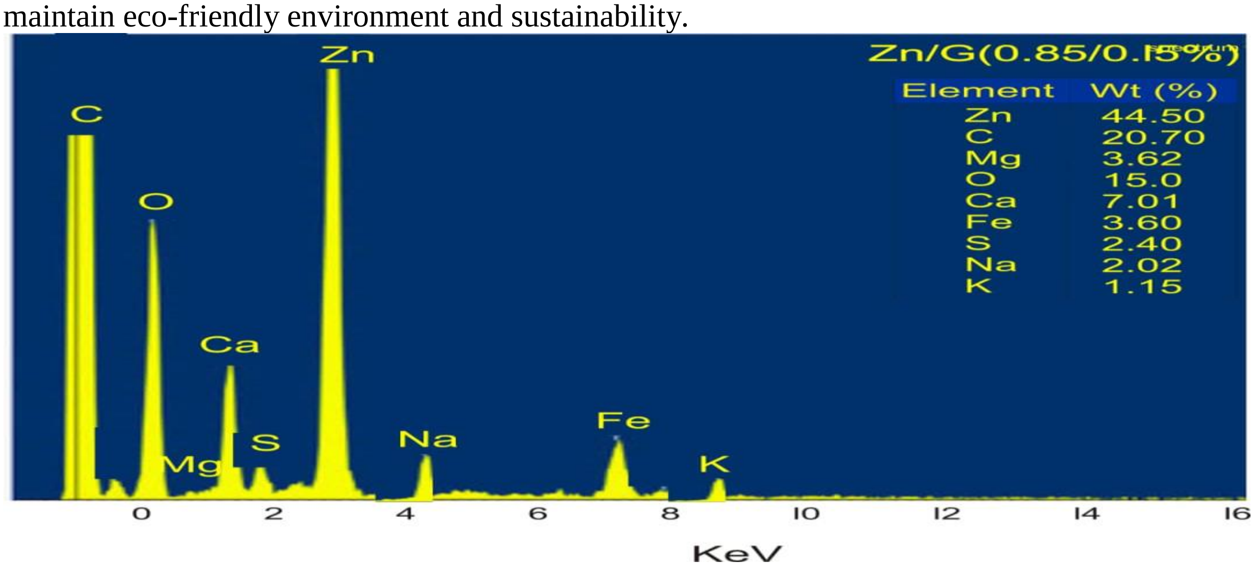

Figure.9: EDX spectrum for (0.85/0.15wt%) zinc-graphene oxide nanocomposite

4. Conclusion

The research achieved green synthesis of zinc–graphene nanocomposites through the application of Manihot esculenta (cassava leaf) extract which served both as a reducing and stabilizing agent. EDX spectroscopy analysis revealed successful incorporation of zinc and graphene oxide into the composite material which comprised 44.50% zinc and additionally included 20.70% carbon and 15.00% oxygen. These findings confirm that a framework of zinc–graphene oxide has formed through bonding between zinc and graphene enabled by oxygen-functional groups. The cassava extract likely produces these elements which strengthen structural stability and boost functional properties through both catalytic and biological mechanisms. Its characteristics ensure suitability for protective coatings applications while also being effective in environmental remediation and biomedical technology. Research into cassava-based nanocomposites through these studies will demonstrate their potential as sustainable nanotechnology materials.

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