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Sustainable Synthesis and Characterization of Graphene Nanosheets from Coconut Husk: A Green Approach for Advanced Materials

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ABSTRACT

Graphene, a two-dimensional carbon material with exceptional mechanical, electrical, and thermal properties, has attracted significant interest for advanced material applications. Conventional production methods often rely on hazardous chemicals and non-renewable resources, creating a need for more sustainable and environmentally friendly approaches. This study explores the environmentally friendly synthesis and characterization of graphene derived from coconut husk waste. This approach contributes to the dual goals of waste valorization and sustainable material development by employing eco-friendly methodologies for graphene production, minimizing the environmental impact compared to traditional methods. Scanning Electron Microscopy (SEM) analysis confirms the successful oxidation of graphite oxide into individual graphene oxide (GO) sheets with a layered and crumpled morphology, suggesting high surface area and self-assembly behavior, both crucial properties for various applications. Raman spectroscopy revealed two dominant peaks in the synthesized GO at 1378.89 cm^{-1} and 1595.89 cm^{-1}, corresponding to the D and G bands, respectively, indicating the presence of both ordered sp^2 carbon and oxygen-induced defects. X-ray Diffraction (XRD) analysis unveiled the crystalline nature of the GO sample, with a prominent peak at $2\theta = 10.13°$ corresponding to the (001) plane, characteristic of oxygen functional groups attached to the GO surface. This research demonstrates the successful conversion of coconut husk waste into high-quality graphene via a sustainable approach and emphasizes the potential for responsible production of advanced materials with diverse applications.

Keywords:
Graphene,
Coconut Husk,
Sustainable Synthesis,
X-ray Diffraction (XRD),
Raman Spectroscopy,
Scanning Electron Microscope (SEM).

INTRODUCTION

In the realm of sustainable materials and technology, researchers are increasingly exploring unconventional sources for the synthesis of advanced materials, aiming to mitigate environmental impact and promote eco-friendly practices. Graphene, a remarkable two-dimensional material known for its exceptional electrical, thermal, and mechanical properties, has garnered significant interest in various industries. Traditionally sourced from graphite, the synthesis of graphene has undergone a paradigm shift towards sustainable approaches, leveraging renewable resources.

Among the diverse array of agricultural wastes, coconut husk has emerged as a promising precursor for graphene synthesis. As a byproduct of the coconut industry, coconut husk represents a vast reservoir of carbonaceous material that can be utilized in the production of high-quality graphene nanosheets (Edwards & Coleman, 2013; Wolf, 2013).

By harnessing the carbon content within coconut husk, researchers can delve into innovative techniques to obtain graphene nanosheets, thereby contributing to both environmental conservation and materials science advancement.

Now, the most popular method for producing single-layered and multi- layered graphene is by solving methods or known as mechanical and chemical methods. For the mechanical method, the graphene produced is single layered.

With the chemical method, graphene produced in large quantities and the preparation of graphene is very simple, but the graphene produced is still not single-layered (Siburian, 2012).

Graphene has proven its potential in various real-world applications. This "Wonder Material" is poised to

revolutionize fields like electronics, energy, and even biomedical technology. For a deeper dive into graphene's applications and future prospects, check out "Science and technology roadmap for graphene..." by Ferrari et al. (2015).

For years, scientists knew about diamond, graphite, nanotubes, and fullerenes - various forms of carbon with diverse properties. But a crucial piece was missing: graphene. Early 2004, at the University of Manchester. A.K. Geim and K.S. Novoselov finally peeled back the mystery, isolating a single layer of carbon atoms - graphene (Novoselov et al., 2004, 2005). Picture splitting graphite, the stuff in pencils, at the atomic level, and you have graphene!This incredible material is just one atom thick, arranged in a honeycomb pattern, and exists in two dimensions. Unlike its hard cousin diamond, graphene is surprisingly soft due to unique vibrations absent in 3D materials. But graphene's true magic lies in its versatility. Graphene synthesis refers to any process of producing or separating graphene from a carbonaceous precursor. Numerous ways have been developed for synthesizing graphene with various desired properties like the unique product's sizes, purity, and efflorescence (Bhuyan et al., 2016). Graphene synthesis can be classified into two major kinds. The top-down approach and the bottom up approach. There are two regularly utilized top-down approaches: mechanical exfoliation of graphite and chemical exfoliation of graphene oxide (chemical technique). In contrast, the most often employed methods for bottom-up approaches include chemical vapor deposition (CVD) and epitaxial thermal development of graphene on substrate. The top-down technique is concerned with separating graphene precursors (graphite) into atomic layers. Graphite is a mono-atomic graphene layer stack held together by weak van der Waals forces. The weak van der Waals pressures must be overcome in order to separate these graphite layers (Edwards and Coleman, 2013). For the chemical technique, the interlayer van der Waals forces can be lowered by oxidizing graphite with a strong oxidizing agent such as concentrated sulfuric acid (H2SO4), sodium nitrate (NaNO3), and potassium permanganate (KMnO4) which produced comparable levels of oxidation and was called as graphene or graphite oxide (GO) based on Brodie method (Brodie,1860), Staudenmaier method (Staudenmaier, 1898), and Hummers method (Hummers and Offeman, 1958). Then, by rapid heating or thermal shock (~1000ºC), the oxidized graphite will then be exfoliated and become single to few layers of graphene oxide (GO) due to the reduction of the interlayer van der Waals forces. The chemical technique, on the other hand, entails sophisticated operations such as numerous chemical treatments that contain harmful compounds. Furthermore, the oxidation treatment will have an effect on the graphene structure as well as medication for weight loss. In the case of mechanical exfoliation, the weak van der Waals forces between mono-atomic graphene layers in graphite were overcome by using external mechanical forces such as scotch tape (Novoselov et al., 2004), ultra-sonication (Ci et al., 2009), electric field (Liang et al, 2008), and transfer printing technique (Liang et al.,2007; Chen et al., 2007).

The production of graphene mono-layers has progressed at an unparalleled rate. Upscaled roll-to-roll fabrication of graphene sheets up to 40 inches wide has been demonstrated (Bae et al., 2010; Vlassiouk et al., 2013). However, this technique is still not suitable for mass production. The bottom-up technique to graphene production implies that carbon precursor atoms grow directly on the substrate surface, giving rise to graphene crystal planes. Thermal vacuum graphitization is used to develop graphene on a single-crystal silicon carbide (SiC) for the epitaxial thermal growth of graphene on substrate method. Sublimation of silicon atoms occurs at roughly 1300 °C under vacuum of SiC because the carbon-enriched surface undergoes reorganization and graphitization. The CVD approach involves the chemical interaction of gaseous carbon precursor molecules that are dispersed on a transition metal substrate at high temperatures (900-1500oC). The solubility of carbon on the substrate decreases as the substrate cools, and the carbon precipitates to produce one to multilayer graphene layers on the substrate.

One of the key advantages of epitaxial and CVD growth techniques is their excellent compatibility with existing CMOS technology due to the ability to manufacture huge areas of high quality graphene. The significant downside of this growth approach is the requirement for expensive substrate materials, high temperatures exceeding 900 to 1500 oC, and the usage of expensive flammable high purity gas for graphene formation, which significantly limits its applications for large-scale production. Recently, researchers proposed and developed green production methods for graphene synthesis that use environmentally friendly biomass resources such as sugar, chitosan, and lucerne plants. The primary goal of green manufacturing is to use less hazardous chemicals and natural precursors (Singh et al., 2017). Green manufacturing, also known as sustainable manufacturing or environmentally conscious manufacturing, is the process of manufacturing products in a way that minimizes environmental impact. This includes using less energy and water, reducing waste, and using less hazardous chemicals. One of the primary goals of green manufacturing is to use less hazardous chemicals and natural precursors. Hazardous chemicals are those that can pose a risk to human health or the environment. They can be toxic, flammable, or corrosive. Natural precursors are raw materials that are derived from nature, such as plants or animals. There are a number of reasons why green manufacturing seeks to reduce the use of hazardous chemicals and natural precursors. First, hazardous

chemicals can pose a risk to the workers who produce them and the consumers who use them. For example, exposure to certain hazardous chemicals can cause cancer, respiratory problems, and skin diseases. Second, hazardous chemicals can pollute the environment if they are not properly disposed of. For example, they can contaminate water and soil, and they can contribute to air pollution. Third, natural precursors are often renewable resources, which means that they can be replenished over time. This makes them more sustainable than non-renewable resources, such as fossil fuels. There are a number of ways to reduce the use of hazardous chemicals and natural precursors in manufacturing. One way is to use safer alternatives. For example, water-based paints can be used instead of solvent-based paints. Another way is to design products and processes that are more efficient. For example, using less material or energy can reduce the amount of waste that is produced. Finally, it is important to properly dispose of hazardous chemicals and waste. This can be done by recycling, incinerating, or landfilling, depending on the type of waste (Edwards & Coleman, 2013; Wolf, 2013; Sunpreet Singh, Ramakrishna, & Gupta, 2017).

Coconut shell charcoal is a natural material with a high carbon content (74.3%) that can be used for the production of graphene oxide (GO) and graphite powder. Several methods have been reported for the graphitization of natural materials, including pyrolysis, template method, foaming technique, and catalytic graphitization. In this study, GO was synthesized from graphite produced from coconut husk waste using a modified Hummers method. Meanwhile, graphite powder was obtained by the graphitization of coconut husk using the pyrolysis method.

MATERIALS AND METHODS

Materials

The starting materials, fresh coconut husks purchased from Gwagwalada market, Gwagwalada Area Council of Abuja, Nigeria. Distilled water, Hydrofluoric acid (HF) 40% solution, Sodium hydroxide (NaOH pellets, Sigma-Aldrich), Potassium permanganate ($KMnO_4$), Sulfuric acid (H_2SO_4), Deionized water.

Synthesis of Graphene Oxide from Coconut Husk Charcoal

This chapter details the methodology employed for the synthesis of graphene oxide (GO) from coconut husk charcoal. The process involved five key steps: pretreatment of coconut husk, combustion to charcoal, purification with hydrofluoric acid (HF), washing and neutralization, and synthesis using the Hummers method.

Pretreatment of Coconut Husk

Fresh coconut husks were collected and thoroughly washed with water to remove any dirt, dust, and adhering particles. The cleaned husks were then dried using a solar thermal treatment for three days. This method was chosen for its low cost and environmental friendliness compared to conventional oven drying.

Combustion of Coconut Husk

After drying, the coconut husks were crushed into granular particles with a size of 2-3 mm using a suitable grinder. The crushed particles were then subjected to combustion in a furnace at 600°C for 3 hours under an inert atmosphere (nitrogen). This process converted the organic components of the husk into high-carbon coconut husk charcoal while minimizing oxidation.

Purification with Hydrofluoric Acid

The obtained coconut husk charcoal was ground and sieved to obtain a fine powder with a particle size of 75 μm using a 200 mesh sieve. This enhanced the surface area and facilitated subsequent treatments. The powder was then washed with 40% hydrofluoric acid (HF) to remove impurities like silica and other inorganic compounds. The acid treatment was carried out at 45°C for 3 hours with a sample-to-acid ratio of 1:3, ensuring efficient purification while minimizing HF consumption.

Washing and Neutralization

Following the HF treatment, the charcoal powder was thoroughly washed with deionized water to remove residual acid and other soluble impurities. The washing process was continued until the pH of the washing solution reached a neutral range (pH 6.7). To complete the neutralization process, the washed powder was further treated with a dilute sodium hydroxide (NaOH) solution and then washed again with deionized water.

Synthesis using Hummers Method

The final step involved the synthesis of GO from the purified coconut husk powder using the modified Hummers method. This well-established method involves oxidation of the carbon structure with strong oxidizing agents like potassium permanganate ($KMnO_4$) and sulfuric acid (H_2SO_4), followed by exfoliation to obtain individual GO sheets. The specific details of the Hummers method modification, including the ratio of reagents, reaction temperature, and duration, need to be adapted based on your chosen protocol and desired GO properties.

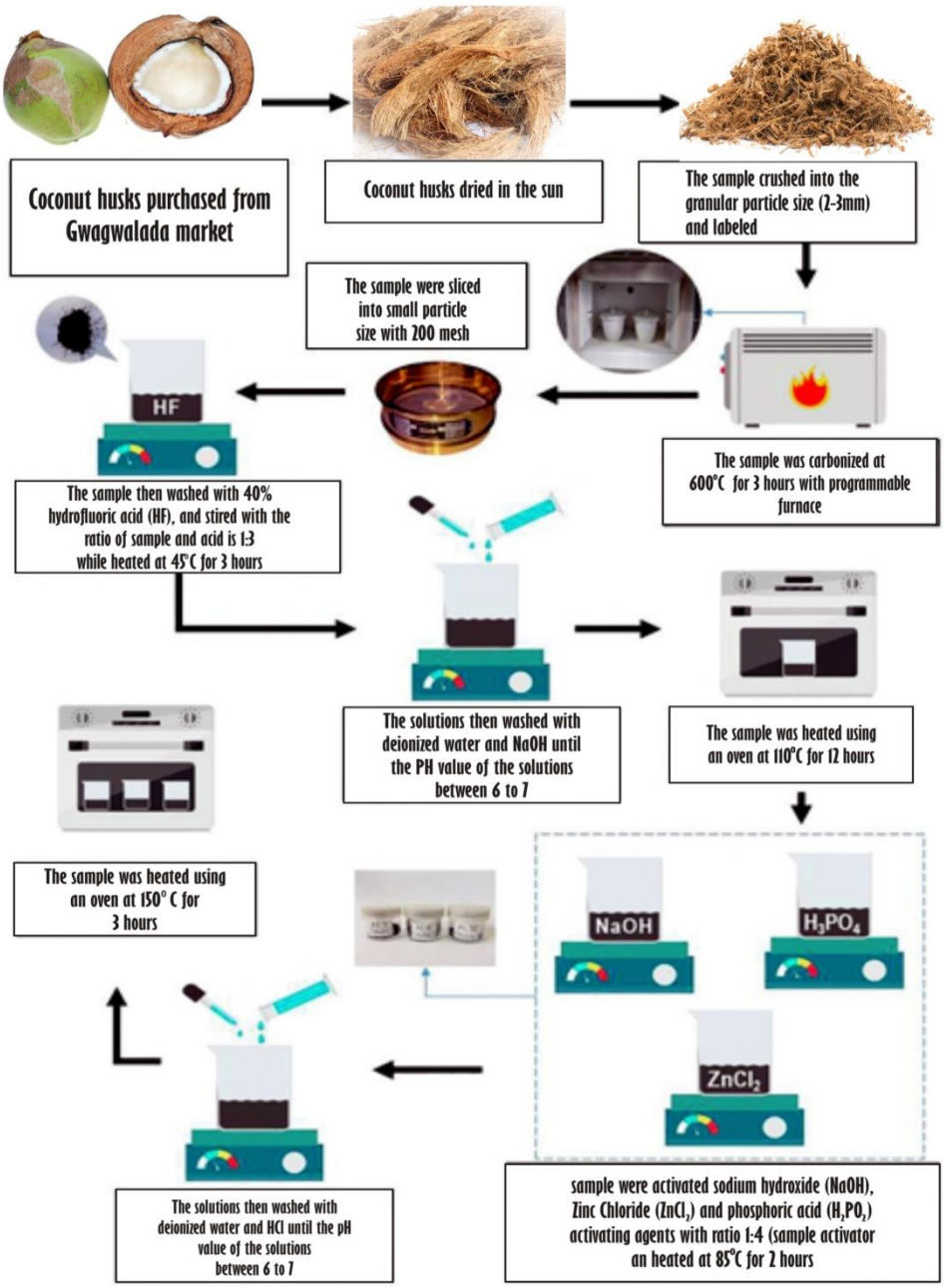

Figure 1: Schematic image of the synthesis process

Characterization

The final product, obtained after purification, was characterized using several advanced analytical techniques to provide a comprehensive understanding of its structural, morphological, and crystallographic properties.

i. Raman spectroscopy: This technique was employed to determine the vibrational modes of the carbon lattice, providing insight into the degree of disorder, defect density, and quality of the graphene sheets. The presence of characteristic D and G bands in the Raman spectra allows differentiation between pristine graphene, graphene oxide, and reduced graphene oxide, while the intensity ratio (ID/IG) indicates the level of structural defects (Zheling Li, Deng, Kinloch, & Young, 2023).

ii. X-ray diffraction (XRD): XRD was used to probe the crystalline structure of the synthesized graphene, providing detailed information on lattice parameters, interlayer spacing, crystal orientation, and phase composition. Peaks corresponding to specific diffraction planes can confirm successful exfoliation of graphite oxide into graphene sheets and reveal the

degree of functionalization (Neakanshika Chadha, Sharma, & Saini, 2021).

iii. Scanning electron microscopy (SEM): SEM provided high-resolution imaging of the graphene's morphology, revealing the sheet-like structure, surface texture, layer stacking, and defects at scales ranging from nanometers to micrometers. This technique is crucial for assessing uniformity, wrinkling, and the presence of agglomerates, which directly affect the material's surface area and potential applications (Edwards & Coleman, 2013; Ivan Vlassiouk et al., 2013).

Collectively, these complementary techniques enabled a thorough evaluation of the structural, morphological, and crystallographic features of the synthesized graphene nanosheets, confirming their high quality and suitability for advanced material applications.

RESULTS AND DISCUSSION

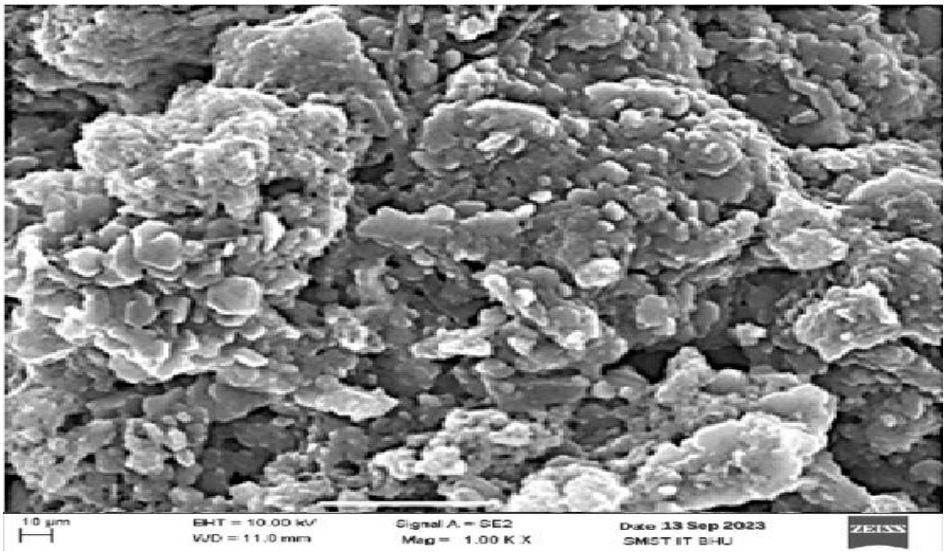

Figure 2: Scanning electron microscopy (SEM) was employed to investigate the surface morphology and structure of the synthesized graphene oxide (GO) nanomaterial

As seen in Figure 2, the SEM image reveals a distinctive surface architecture characterized by two key features:

Flat surface with ordered layer structure: The image showcases a relatively flat surface composed of layered structures, indicating the presence of stacked GO sheets. This observation suggests the successful exfoliation of graphite oxide into individual GO sheets, maintaining their inherent layered arrangement.

Randomly aggregated thin, crumpled layers: Closer examination reveals the presence of numerous thin, crumpled GO layers randomly aggregated throughout the image.

This wrinkled morphology is a typical characteristic of GO due to its high surface area and tendency to self-assemble.

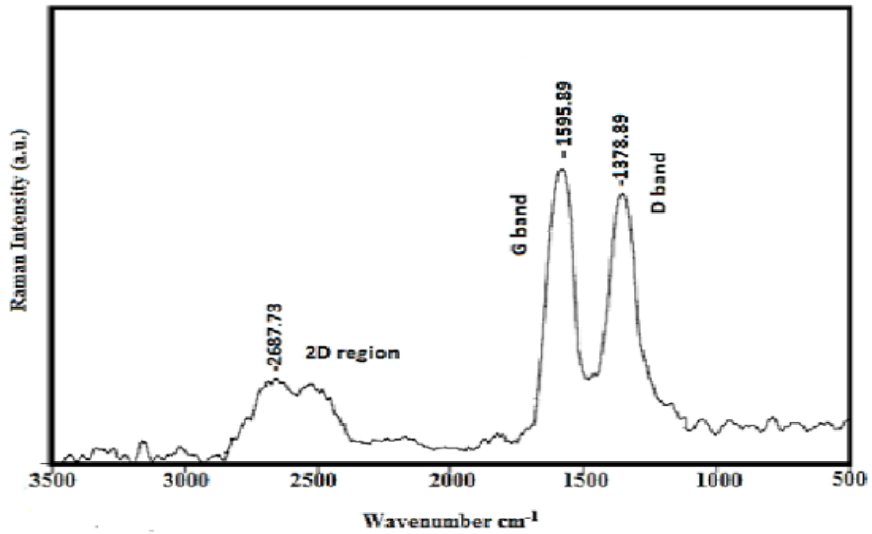

Figure 3: Raman spectrum of the synthesized graphene

Figure 3 unveils the Raman spectrum of the synthesized graphene oxide (GO), offering a unique molecular fingerprint. Two prominent peaks dominate the spectrum, located at 1378.89 cm^{-1} and 1595.89 cm^{-1}, respectively. Notably, Raman spectroscopy serves as a powerful tool for estimating the number of GO layers in our sample (Z. Li et al, 2023).

These dominant peaks belong to the D band (1378.89 cm^{-1}) and the G band (1595.89 cm^{-1}). The D band arises from local defects and disordered structures existing at the edges of GO sheets, while the G band signifies the characteristic sp^2 carbon hybridization within the observed multilayered stacks. The ratio between their intensities (ID/IG) provides valuable insights into the material's quality (Chadha et al, 2021).

In our case, the ID/IG ratio is calculated as 0.86, reflecting a prominent D band. This observation points towards the presence of significant structural imperfections within the GO sheets, likely induced by the attachment of hydroxyl and epoxide groups during the oxidation process.

Furthermore, a less intense peak emerges at 2687.73 cm^{-1}, corresponding to the 2D band. While less prominent, this peak plays a crucial role in determining the number and orientation of GO layers within the sample.

In essence, the Raman spectrum paints a captivating picture of the GO's molecular structure, revealing both the ordered sp^2 carbon framework and the presence of defects arising from oxygen functionalization. This information is crucial for understanding the overall quality and properties of the synthesized GO.

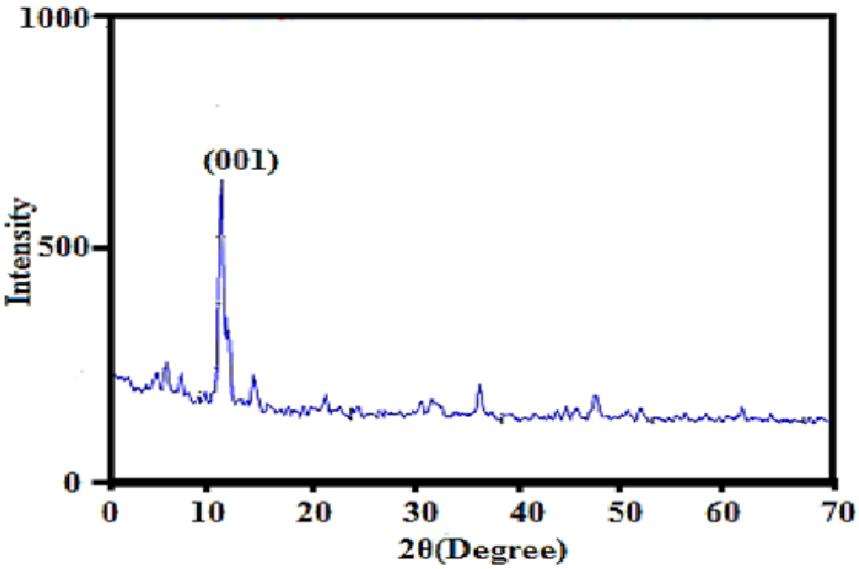

Figure 4: X-Ray Diffraction

Figure 4 presents the XRD pattern of the synthesized graphene oxide (GO) sample, offering insights into its crystalline structure. The analysis reveals a distinct peak at 2θ = 10.13°, corresponding to the (001) plane of GO. This specific peak indicates an increase in the interlayer spacing compared to pristine graphite, likely due to the presence of oxygen-containing functional groups on the GO surface, as reported in previous studies.

CONCLUSION

This study demonstrates the successful transformation of coconut husk, an abundant agricultural waste, into high-quality graphene via an environmentally friendly and sustainable synthesis method. The work highlights the potential of valorizing agricultural waste into valuable materials, thereby reducing environmental impact and contributing to green manufacturing. By utilizing readily available coconut husk, this approach offers a promising route for scalable and eco-friendly production of graphene for a wide range of applications, including electronics, energy storage, and catalysis. Characterization of the synthesized graphene oxide (GO) confirmed its high quality and structural integrity. Scanning Electron Microscopy (SEM) revealed the formation of individual GO sheets with a layered, crumpled morphology, indicative of a high surface area and self-assembly behavior—properties essential for diverse applications. Raman spectroscopy showed prominent D and G bands at 1378.89 cm^{-1} and 1595.89 cm^{-1}, respectively, reflecting both the ordered sp^2 carbon framework and oxygen-induced defects within the GO structure. X-ray diffraction (XRD) analysis further confirmed the crystalline nature of the material, with a prominent peak at 2θ = 10.13° corresponding to the (001) plane, indicating expanded interlayer spacing due to oxygen-containing functional groups. Overall, this research not only demonstrates the effective conversion of coconut husk waste into high-quality graphene through a sustainable approach but also underscores the potential of such green methodologies for the responsible and scalable production of advanced materials with multifunctional applications.

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