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Graphene-Based Electrode Materials via Liquid Phase Exfoliation from Chicken Feather Waste

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ABSTRAK

Graphene was successfully synthesized from chicken feather biomass waste using the Liquid Phase Exfoliation (LPE) method assisted by Linear Alkylbenzene Sulfonate (LAS) surfactant at varying volumes of 1 mL, 1.5 mL, and 2 mL. The aim of this study was to evaluate the potential of chicken feather-derived graphene as an electrode material for rechargeable batteries. Characterization using UV-Visible spectrophotometry showed a distinct absorption peak at approximately 270 nm, corresponding to the $\pi \rightarrow \pi^*$ electronic transition, with increasing absorbance intensity observed with higher surfactant volumes, indicating more effective graphene exfoliation. XRD analysis revealed a shift in diffraction peaks to lower 2θ angles, increased interlayer spacing (d-spacing), and the largest crystallite size of 13.58 nm at a surfactant volume of 1.5 mL. These results indicate that the surfactant plays a critical role in stabilizing and forming the crystalline structure of graphene. Overall, this study demonstrates that chicken feather-based graphene has promising potential to be developed as an eco-friendly electrode material in rechargeable battery application.

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I. INTRODUCTION

Over the past few decades, carbon-based materials-particularly graphene-have become a major focus in energy storage research. Graphene is known for its exceptional properties, including high electrical conductivity, large specific surface area, strong mechanical strength, high charge mobility and outstanding chemical stability (K. S. Novoselov et al, 2016)(Su & Hu, 2021). These combined characteristics make graphene one of the most promising materials for enhancing the performance of various rechargeable batteries, such as lithium-ion batteries, supercapacitors, and next-generation battery technologies (Zhang & Zhao, 2009).

In the last decade, research on graphene for electrode materials has grown significantly, as has been done by Devendran & Nagai. (2023) who discuss the latest developments in the design and application of three-dimensional (3D) porous graphene-based electrodes for electrochemical energy storage systems, such as lithium ion batteries (LIBs), lithium-sulfur batteries (LSBs), sodium ion batteries (SIBs), and supercapacitors. In addition, (Mangesh, 2025) also discusses the potential of graphene as an electrode material in next-generation supercapacitors, which are capable of fast charging, high power, and long service life.

However, conventional methods of graphene production, such as chemical exfoliation and chemical vapor deposition (CVD), still face significant challenges, especially in terms of high costs, scalability, and environmental impact (Zhu et al., 2010). Therefore, exploring alternative, more sustainable raw material sources for graphene is crucial.

One emerging approach being developed is the synthesis of graphene from biomass waste, including chicken feathers. This type of waste, which is abundantly available, contains keratin, a protein rich in carbon and nitrogen that has great potential to be converted into carbon-based materials like graphene (Li et al., 2024).

Utilizing chicken feather-derived graphene in rechargeable battery applications offers the potential to create high-performance batteries with longer life cycles, faster charging rates, and lower production costs. Moreover, this strategy aligns with the principles of a circular economy by transforming food industry waste into high-value products in the energy sector (Imawati et al., 2024).

Graphene can be synthesized through two main strategies, top-down and bottom-up approaches. The top-down method entails the exfoliation of graphite layers to produce individual graphene sheets, while the bottom-up approach involves constructing graphene from various carbon-containing precursors, as seen figure 1 (Edwards & Coleman, 2013).

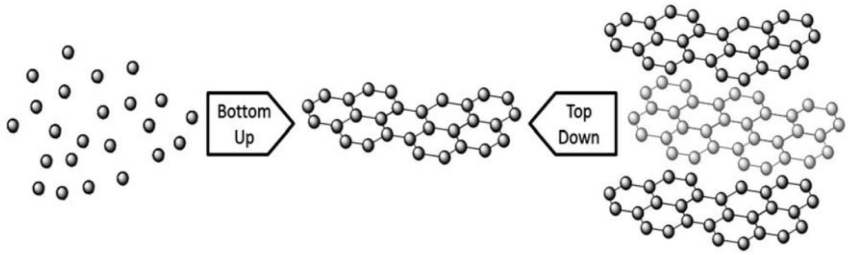


Figure 1 Schematic illustration of graphene synthesis via bottom-up and top-down approaches

The bottom-up approach produces graphene with fewer surface defects compared to the top-down method. However, it is relatively more complex and requires higher production costs. In contrast, the top-down approach is more suitable for large-scale graphene production due to abundant raw materials, no special equipment required, its simpler and more straightforward process (Liu et al., 2020)(Ghatule & Devare, 2015). Commonly used top-down methods include liquid-phase exfoliation and micromechanical cleavage of graphite (Al-Dhahebi et al., 2020). This approach is widely applied for producing graphene in large quantities, where the resulting graphene nanosheets can be easily combined with functional components to develop new materials. The most frequently employed top-down technique is oxidative exfoliation of graphite (G. Zhao et al., 2012).

In this study, graphene was synthesized from chicken feathers using the Liquid Phase Exfoliation (LPE) method, a relatively simple and cost-effective technique for producing graphene on a large scale (Hernandez et al., 2008). This process involves exfoliating carbon materials in a solvent using shear force or ultrasonic waves. The resulting graphene was then tested for application as an

electrode material in rechargeable batteries. The aim of this research is to assess the potential of chicken feather-based graphene as an electrode material, thereby promoting the use of biomass waste in the development of sustainable energy technologies.

II. METHOD

2.1 Preparation of Graphite

Graphite powder was synthesized from chicken feather waste through a carbonization process. Initially, the chicken feathers were thoroughly washed to remove any impurities and then dried in an oven at 200 °C for 2.5 hours. The dried feathers were subsequently carbonized in a furnace at 400 °C for 2 hours. The resulting carbonized material was ground into fine powder and characterized using X-ray Diffraction (XRD) to confirm the formation of graphite.

2.2 Preparation of Graphene

To synthesize graphene, 2 gr of the prepared graphite powder were dispersed in 250 mL of deionized water along with varying volumes (1.0, 1.5, and 2.0 mL) of Linear Alkylbenzene Sulfonate (LAS) as a surfactant. The mixture was continuously stirred at 75 °C for 30 minutes to ensure uniform dispersion. Following this, the solution was subjected to ultrasonication for 5 hours to exfoliate the graphite. After sonication, the suspension was allowed to stand undisturbed for 72 hours to facilitate sedimentation. The resulting precipitate was collected, washed with deionized water to remove residual surfactant, and dried at 100 °C for 2 hours to obtain graphene powder.

2.3 Characterization of Graphene

The crystalline structure of carbon-based materials (graphite and graphene) was characterized using X-ray diffraction (X'Pert PRO, PANalytical MPD PW3040/60) with Cu Kα radiation, and from the resulting data were plotted and determined FWHM value using Origin 2024 software. Determination of the crystallite size of graphene using the Scherrer equation such as Equation (1).

$$L = \frac{K\lambda}{B\cos\theta} \tag{1}$$

where, L is crystallite size (nm), K is Scherrer constant (typically 0.9), λ is X-ray wavelength (for Cu Kα = 0.15406 nm), B is Full Width at Half Maximum (FWHM) of the diffraction peak (radians), θ is Bragg angle in radians, which is half of 2θ. For the optical properties and absorbance peaks of the graphene samples were investigated using UV–Visible spectrophotometry (T70 spectrophotometer) and the resulting data were plotted using Origin 2024 software.

III. RESULT AND DISCUSSION

3.1 UV-Visible Analysis

UV-Visible testing was carried out to analyze the electron structure and confirm the presence of graphene synthesized using surfactants with varying volumes of 1 mL, 1.5 mL, and 2 mL. The results of the UV-Vis spectrum are shown in Figure 2 in the form of a graph showing the relationship between absorbance value and wavelength.

At concentration 1 mL exhibited a prominent absorbance peak at approximately 284 nm, corresponding to the characteristic π→π* electronic transition of C=C bonds in the sp^2-hybridized carbon structure of graphene, in agreement with previously reported studies (Ferrari et al., 2006). At concentrations of 1.5 mL and 2 mL there was a shift in the absorption peak at approximately 297 nm indicating a change in the electronic structure of graphene. The enhanced absorbance indicates a greater concentration of dispersed graphene in the solution, suggesting more effective exfoliation. The surfactant plays a vital role in stabilizing the exfoliated graphene sheets by preventing re-agglomeration, likely through steric hindrance or electrostatic repulsion mechanisms. Research by Mindivan et al. (2021) showed that the use of cetyltrimethylammonium bromide (CTAB) surfactant at a certain concentration can increase the stability of graphene dispersion, but too high a concentration can cause a decrease in exfoliation efficiency due to excessive micelle formation.

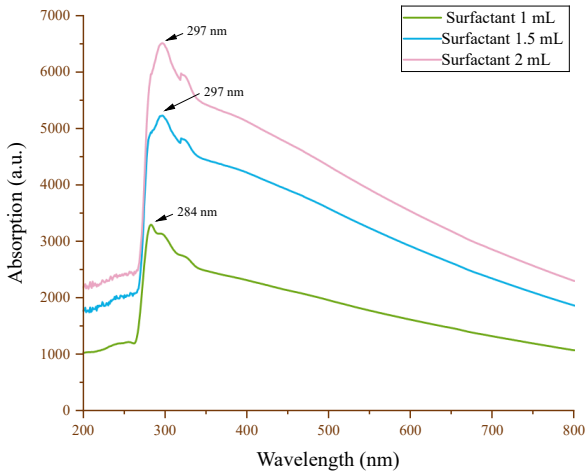


Figure 2 UV-Visible spectrum of graphene synthesized using varying surfactant volumes

Additionally, the appearance of a shoulder at higher wavelengths may suggest the presence of residual oxygen-containing functional groups, such as hydroxyl (-OH) or carbonyl (C=O), which were not entirely removed during the exfoliation process. Although commonly regarded as defects, these functional groups can be advantageous in battery applications, potentially enhancing lithium-ion interaction and improving cycle performance (Zhu et al., 2010). The Liquid Phase Exfoliation (LPE) method employed in this study demonstrated its effectiveness in producing graphene with well-maintained π-conjugated structures, without requiring harsh chemical treatments. This highlights the feasibility of a sustainable, biomass-based approach to graphene synthesis using chicken feather waste (Parvez et al., 2014).

In summary, increasing the surfactant concentration from 1 mL to 1.5 mL and 2 mL, then increased the efficiency of graphene exfoliation, resulting in better dispersion and changes in the electronic structure reflected in the shift of the UV-Vis absorption peak. However, it is important to note that too high a surfactant concentration can cause the formation of micelles which actually inhibit the exfoliation process. Therefore, optimization of the surfactant concentration is very important to produce graphene with optimal optical and structural qualities.

3.2 X-Ray Diffraction Analysis

X-ray diffraction (XRD) analysis of graphite and exfoliated materials prepared using various volumes of surfactant revealed significant changes in diffraction patterns as seen Figure 3. For the pure graphite sample, the primary diffraction peak appeared at 2θ = 21.4°, which corresponds to a tetragonal crystal structure of graphite with a d-spacing value of 4.15 Å. According to research by Santika et al. (2023), graphite synthesis using chicken feathers produces optimum results at a carbonization temperature of 400 ºC.

Following the exfoliation process using surfactants, the main peak shifted toward lower 2θ angles. The sample prepared with 1 mL of surfactant exhibited a peak at 2θ = 25.78°, while the use of 1.5 mL and 2 mL surfactants resulted in peaks at 25.44° and 25.37°, respectively. These shifts indicate an increase in interlayer spacing (d-spacing) from 3.35 Å to 3.44 Å, 3.46 Å, and 3.48 Å, respectively. The increased d -spacing suggests that surfactant molecules were intercalated between graphite layers, disrupting the structural order and expanding the interplanar distance (Kartick et al., 2013).

Additionally, a reduction in diffraction peak intensity was observed in the exfoliated samples compared to pure graphite. This reduction indicates a decrease in crystallinity due to exfoliation, resulting in a more amorphous material with more separated layers (Taufantri et al., 2016) (Johra et al., 2014). These findings suggest that the surfactant-assisted exfoliation process not only increases interlayer spacing but also reduces the crystallinity of the structure.

Furthermore, at a surfactant concentration of 1 mL, several diffraction peaks in the XRD pattern have not appeared or appear weak compared to concentrations of 1.5 mL and 2 mL. This is due to the amount of surfactant used is not sufficient to support the exfoliation process of graphite into graphene effectively. Research by Luo et al. (2020) shows that increasing the concentration of surfactants can

increase the efficiency of graphene exfoliation, resulting in a more homogeneous dispersion and higher graphene concentration. However, there is an optimal limit, because too high a surfactant concentration can cause the formation of micelles which actually inhibit the exfoliation process.

Nevertheless, at a surfactant concentration of 1 mL, graphene exfoliation is not optimal, which causes several diffraction peaks in the XRD pattern to not grow or appear weak. Increasing the surfactant concentration to 1.5 mL or 2 mL increases the exfoliation efficiency, resulting in better graphene dispersion and clearer XRD diffraction peaks.

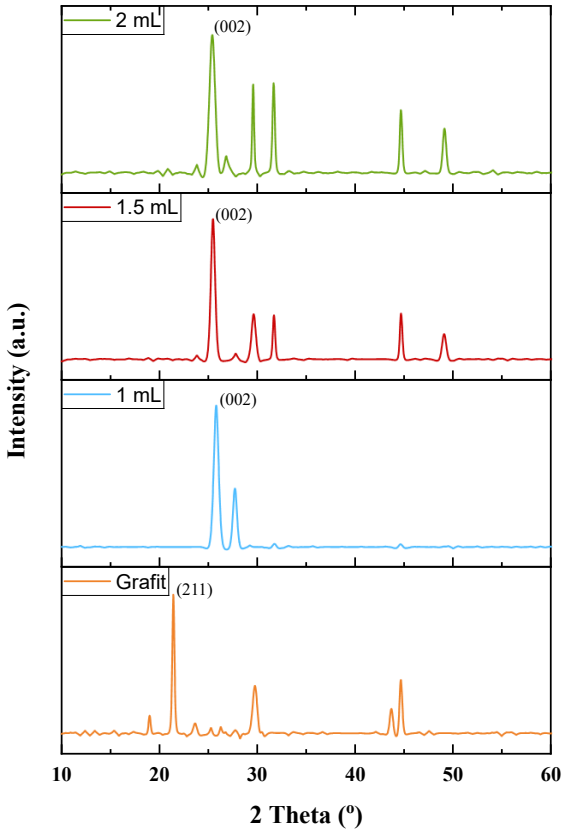


Figure 3 XRD pattern of graphite and graphene synthesized using varying surfactant volumes

The variation in surfactant volume (1 mL, 1.5 mL, and 2 mL) significantly affects the diffraction peak position and full width at half maximum (FWHM), which directly influence the calculated crystallite size based on the Scherrer equation as seen Table 1. Among the samples, the use of 1.5 mL surfactant yielded the bigger crystallite size of 13.58 nm, than 1 mL (11.97 nm) and 2 mL (10.49 nm). These results indicate that the amount of surfactant plays a crucial role in the formation of graphene crystalline structures. An optimal surfactant concentration helps stabilize graphene sheets during the exfoliation process, resulting in larger and more ordered crystals.

Table 1. Crystallite size of graphene at different surfactant volumes

Surfactant Volumes	θ (rad)	FWHM (rad)	L (nm)
1mL	0.2249	0.01017	11.97
1.5 mL	0.2221	0.00881	13.58
2 mL	0.2214	0.01146	10.49

Surfactants help to reduce the interfacial tension between the solvent and graphene and support efficient dispersion of graphene sheets. However, at high concentrations (2 mL), the formation of micelles or aggregates can hinder uniform crystal growth. Conversely, too low a concentration (1 mL) can result in instability of graphene sheets during the exfoliation process, resulting in smaller crystal

sizes. This is in line with the research of (Wang et al., 2020) which showed that increasing surfactant concentration is only effective up to a certain limit.

In addition, a narrower FWHM value indicates a larger crystallite size, in accordance with the basic principle of XRD that FWHM is inversely proportional to crystallite size (Scherrer). This relationship is also strengthened by the findings of (Zhao et al., 2011) which showed a relationship between FWHM and graphene oxide crystallite size based on synthesis parameters.

The use of surfactants has a significant impact on the structural transformation of graphite. Increasing the surfactant volume leads to a greater interlayer spacing, indicating that surfactants not only function as anti-agglomeration agents but also intercalate between graphite layers, thereby expanding the d-spacing and promoting the formation of graphene or thin-layered graphite materials. However, based on the results, a volume of 1.5 mL was found to be the optimal condition for producing graphene with larger crystallite size and a more ordered structure. This is particularly important for applications such as electrode materials and electrical conductors, where larger crystallites help reduce grain boundaries and structural defects, thus enhancing conductivity.

IV. CONCLUSIONS

The characterization results show that the quality and structure of the graphene produced are greatly influenced by the amount of surfactant used during the exfoliation process. UV-Vis characterization shows that all samples show a typical graphene absorption peak at around 270 nm, with the highest absorbance intensity in the sample with a surfactant variation of 2 mL indicating the largest amount of dispersed graphene. However, XRD characterization shows that the largest crystal size is obtained in the sample with a surfactant variation of 1.5 mL, which is 13.58 nm, indicating optimum exfoliation without excessive damage to the graphene crystal structure. So it can be concluded that a surfactant volume of 1.5 mL is the optimum condition for the synthesis of graphene from chicken feathers which shows a good crystal structure, large crystal size, and optical characteristics suitable for application as an electrode material in rechargeable batteries. This study also confirms that biomass waste such as chicken feathers can be utilized effectively to produce high-value functional materials, in line with the sustainable energy and circular economy approaches.

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