



Synthesis and physical characterization of lithium-ion battery anode with silicon-graphene-aluminum oxide composite

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ABSTRACT

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Extensive research has focused on advanced anode materials to overcome the capacity limitations of traditional graphite anodes and meet the growing demand for high-energy-density lithium-ion batteries. Composite anodes have gained increasing attention due to their ability to combine the properties of the constituent materials. This combination can improve electrical conductivity, mechanical stability, electrochemical performance, and cycling stability by overcoming the limitations of individual anode materials. To improve energy-storage applications, this article aims to combine the individual properties of silicon, graphene and aluminum oxide. Silicon–graphene–aluminum oxide (Si–G–Al₂O₃) composite anodes were fabricated via a scalable, straightforward slurry-based process. Graphene was incorporated to enhance electrical conductivity and structural flexibility, whereas Al₂O₃ was used to improve interfacial stability and mechanical robustness of the fabricated anodes. Three composite samples were prepared with different amounts of each material by accurately weighing the constituents: S1 (94% graphene, 5% silicon, 1% Al₂O₃), S2 (84% graphene, 15% silicon, 1% Al₂O₃), and S3 (89% graphene, 10% silicon, 1% Al₂O₃). The mixtures were processed by mechanical mixing and ultrasonic dispersion, with polyacrylic acid (PAA) as a binder to improve mechanical integrity and interfacial adhesion. The morphology, chemical properties, and structural integrity of the fabricated samples were characterized using Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FTIR) spectroscopy, and X-ray Diffraction (XRD). The SEM, FTIR, and XRD results indicate that the composites were successfully formed and maintained good phase integrity, making them viable anode candidates for high-performance lithium-ion batteries.

Contribution/Originality: This study contributes by developing a silicon–graphene–aluminum oxide composite anode, combining the high lithium-storage capacity of silicon with the enhanced conductivity of graphene and the structural stability of aluminum oxide, whereas most of the existing studies have focused on binary Si-based composites.

1. INTRODUCTION

Increased economic growth and rapid urbanization have directed the modern world towards significant energy consumption. The increasing trend of transportation is also liable for this excess energy demand. In contrast, limited

fossil fuel resources and their environmental impact are a major concern in meeting this demand. Fossil fuels contribute to greenhouse gas emissions, particularly carbon dioxide (CO₂), which is liable for exacerbating global warming as well as climate change, which has heightened the significance of alternative energy sources. Consequently, pollution levels are rising globally, and energy demand is expected to grow significantly in industrialized nations. It is thus essential to accentuate renewable and environmentally friendly energy sources for a sustainable future (Alex et al., 2026). Among renewable energy sources, solar, hydropower and wind are the most widely used sources due to their sustainable nature. These sources, however, are not sufficient to meet the needs of an efficient energy storage system due to their intermittent nature.

Battery technology has become indispensable for electric and hybrid vehicles, as well as portable electronic devices such as laptops, remote control devices, mobile phones, and drones, with the advent of recent advances in electric and hybrid vehicles (Ali et al., 2023; Ruby Raj, Lee, Reddy, & Zaghib, 2024). Currently, lithium-ion batteries are widely used predominantly because of their impeccable performance, better energy density, long cycle life, and extraordinary electrochemical characteristics unmatched by any other type of battery (Ma et al., 2025; Tunca et al., 2024). It also has a high operating voltage and comparatively low self-discharge rate. However, the battery in EVs cannot meet the required driving range when it needs to maintain a long service life (Choi & Aurbach, 2016). Therefore, Li-ion batteries may need to be improved to provide adequate energy and power density, a long cycle life, and low manufacturing costs, as well as minimal environmental impact (Barcaro, Marangon, Mutarelli, & Hassoun, 2024).

To meet the high energy demand, researchers are conducting ongoing research on anode and cathode materials that may contain many more active materials, such as the oxidation/reduction (redox) reactions between the battery anode and cathode, which largely depend on the amount of active material present in each electrode (Mahmud et al., 2022). This research focuses on the fundamental battery components, such as the anode, cathode, electrolyte, and separator. The oxidation/reduction (redox) reactions that occur between the battery electrodes depend heavily on the amount of active material in each electrode (Yerkinbekova et al., 2025). The traditional graphite anodes used in lithium-ion batteries have a low energy capacity (372 mheavilyAh/g), which ultimately decreases the overall energy density (Asenbauer et al., 2020). However, the huge demands of modern battery applications require high capacity and improved electrochemical performance. In particular, large-scale energy storage and electric vehicles need batteries that are capable of storing more energy with cyclic stability during charging and discharging. Batteries with higher energy-storage capacity can significantly increase the driving range of electric vehicles. A higher-capacity battery can store more electrical energy within the same charging cycle, which allows the vehicle to travel long distances before requiring recharging.

Silicon anodes are among the most favourable next-generation energy storage material systems owing to their high theoretical capacity of 4200 mAh/g, which is 10 times that of traditional graphite and low discharge potential (< 0.4 V) (Feyzi et al., 2024; Sun et al., 2022). However, during repeated lithiation and delithiation cycles, silicon undergoes significant volume changes up to 300%, which exacerbates performance as a battery electrode (Feng et al., 2018; Jhan, Wang, Sung, & Tzeng, 2024). Also, the intrinsic electronic conductivity of Si is only about 10⁻⁴ S/m, and the 30-fold volume expansion compared with commercial graphite anodes at the end of the lithiation process limits its direct use as an anode (Shi et al., 2024; Wang et al., 2023). These cause stress introduction, mechanical damage, capacity loss, and formation of a solid electrolyte interphase (SEI) (Ge et al., 2021; Zhao et al., 2023). The proposed strategies for enhancing silicon (Si) materials during lithiation include reducing Si particle size to mitigate mechanical stress; developing coatings to reinforce the Si microstructure and limit excessive solid-electrolyte interphase (SEI) deposition; employing chemical additives in the electrolyte to stabilize the SEI; and integrating Si with highly conductive materials (Gu et al., 2022; Han et al., 2022; Kim et al., 2017; Lang et al., 2017; Zhang, Wang, Pushparaj, Mann, & Hou, 2022).

There has been extensive research on the physics of graphene in energy storage as well as other research arenas owing to its very high specific surface area, high thermal and electrical conductivity, and exceptional strength, making it the most-studied single-layer form of carbon. The limitations of silicon particles can be mitigated by incorporating graphene, which can provide sufficient electrical conductivity and structural rigidity. A larger amount of electricity can also be stored by graphene than by traditional graphite, which has an energy density of around 744 mAh/g when converted to Li_2C_6 (Lian et al., 2010).

Recent literature has shown that the combination of silicon and carbonaceous materials such as graphene oxide (GO) and reduced graphene oxide (rGO) can alleviate the associated shortcomings of silicon anodes. Zhou et al. (2025) fabricated a graphene/silicon/carbon composite where nano silicon was directly incorporated onto graphene and then coated with hard carbon. The extreme volume expansion of silicon caused rapid capacity fade, thus highlighting the importance of using a carbon coating in their research to achieve high capacity, improved conductivity, along with mechanical and interfacial integrity. Another study (Karimi, Sadeghi, & Ghaffarinejad, 2026) investigated the direct combination of silicon and rGO, where Si nanoparticles were uniformly anchored and partially encapsulated by rGO sheets, which produced a continuous conductive network around the Si particles. The flexible nature of rGO ensured lower volume change of silicon as well as maintained proper electrical conductivity. A further study (Lee, Kim, Byon, Choi, & Jung, 2026) reported that nano-sized silicon with both GO and rGO can enhance the structural integrity as a lithium battery anode. They reported that the incorporation of GO is responsible for better dispersion and structural integrity, whereas rGO is responsible for better electronic conductivity.

Aluminum oxide, a ceramic-based material, is extensively used to protect batteries, as it is chemically inert and thermally stable, thereby helping maintain structural integrity. Recent research has shown that Al_2O_3 can create functional surfaces for lithium-ion transport, thereby facilitating ion transport and preventing undesired side reactions (Zhou et al., 2023). There is also evidence that Al_2O_3 incorporation can prevent undesired side reactions between the electrode and the electrolyte in lithium-ion batteries while still ensuring that lithium ions can move through (Friesen et al., 2017).

A different strategy (Azevedo Costa, Esken, Gambaryan-Roisman, & Menzel, 2025) investigated Al_2O_3 -coated SiO_x/C particles. The study found that a relatively low proportion of Al_2O_3 can significantly modify the Si-electrolyte interface. Among the various compositions, 1 wt.% Al_2O_3 delivered the best performance. This is because lower Al_2O_3 loadings provided insufficient protection, whereas excessive Al_2O_3 exacerbated the interfacial resistance and reduced transport. Additionally, Al_2O_3 acted as a hydrofluoric (HF) scavenger, forming an AlF_3 -containing passivation layer that hindered undesirable electrolyte reactions at the Si surface. Therefore, Al_2O_3 primarily improves the chemical and mechanical stability of the interface rather than supplying electronic conductivity. The practical performance of silicon-based lithium-ion batteries can be improved using an Al_2O_3 -modified Si/C microparticle anode (Lv et al., 2025). Hard carbon was employed to modify the particle size, while soft carbon was incorporated to enhance electrical conductivity. Additionally, the incorporation of Al_2O_3 formed a thin solid electrolyte interphase, which greatly maintained the electrode stability. The prepared anode with an NMC cathode-based full cell displayed excellent performance.

Although extensive research has been conducted on individual components, less investigation has examined the compositions of silicon-graphene-aluminum oxide composite anodes. The combination of these three materials can mitigate the associated failure modes of silicon, such as volume change, unstable SEI, and interfacial degradation, by creating a more balanced high-capacity anode. Graphene can offer an elastic conductive structure to accommodate Si expansion as well as ensure electronic contact, while Al_2O_3 can offer high mechanical and interfacial stabilization by inhibiting side reactions and generating a more stable SEI. The combination thus presents a potentially synergistic pathway to higher-capacity, longer cycle life silicon-based lithium-ion battery anodes. Due to this, this research aims to synthesize and characterize silicon-graphene-aluminum oxide composite anodes with three different compositions

and then analyze their morphological, structural, and chemical properties using SEM, XRD, and FTIR to identify potential high-performance lithium-ion battery anodes.

2. MATERIALS AND METHODOLOGY

2.1. Materials

Here, three samples were made employing graphene, silicon, aluminum oxide and Polyacrylic Acid (PAA) binder. Graphene (Gr) nanopowder (purity 95%, thickness 5–10 nm; CAS No. 7782-42-5, molecular weight 12.01; supplied by Rajpalya, Hoodi, Bangalore-560048, India). Silicon (Si) powder (200 mesh, 98.5% extra pure; MF-Si, molecular weight 28.09, assay $\geq 98.5\%$; Mumbai, India). Aluminum Oxide (Al_2O_3) was collected from Loba Chemie Pvt. Ltd. (India) for its chemical stability and thermal resistance, which protect the cathode surface and enhance stability. Polyacrylic Acid (PAA), with the chemical formula $(\text{C}_3\text{H}_4\text{O}_2)_n$ was purchased from Sigma-Aldrich (Merck) and used as a binder in electrode fabrication, which provides chemical stability as well as structural integrity.

2.2. Sample Preparation

In this study, three composite anode samples were prepared with different weight percentages of graphene and silicon. The amount of aluminum oxide was kept constant for all three samples. The composition of each sample was selected according to the proportions listed in Table 1. Before fabrication, each material was weighed separately and accurately. A weighing balance was used to ensure that the required amount of each component was obtained. The measured quantities of graphene, silicon, and aluminum oxide were then used to prepare the corresponding composite anodes. This process allowed the effect of changing the graphene and silicon proportions on the properties of the composite anodes to be investigated while keeping the amount of aluminum oxide constant.

Table 1. Details about the composition of the fabrication samples.

Sample No.	Composition	Weight of the composition		
		Graphene (gm)	Silicon (gm)	Al_2O_3 (gm)
S1	94% Graphene, 5% Si, and 1% Al_2O_3	0.988	0.052	0.010
S2	84% Graphene, 15% Si, and 1% Al_2O_3	1.000	0.178	0.012
S3	89% Graphene, 10% Si, and 1% Al_2O_3	0.988	0.111	0.011

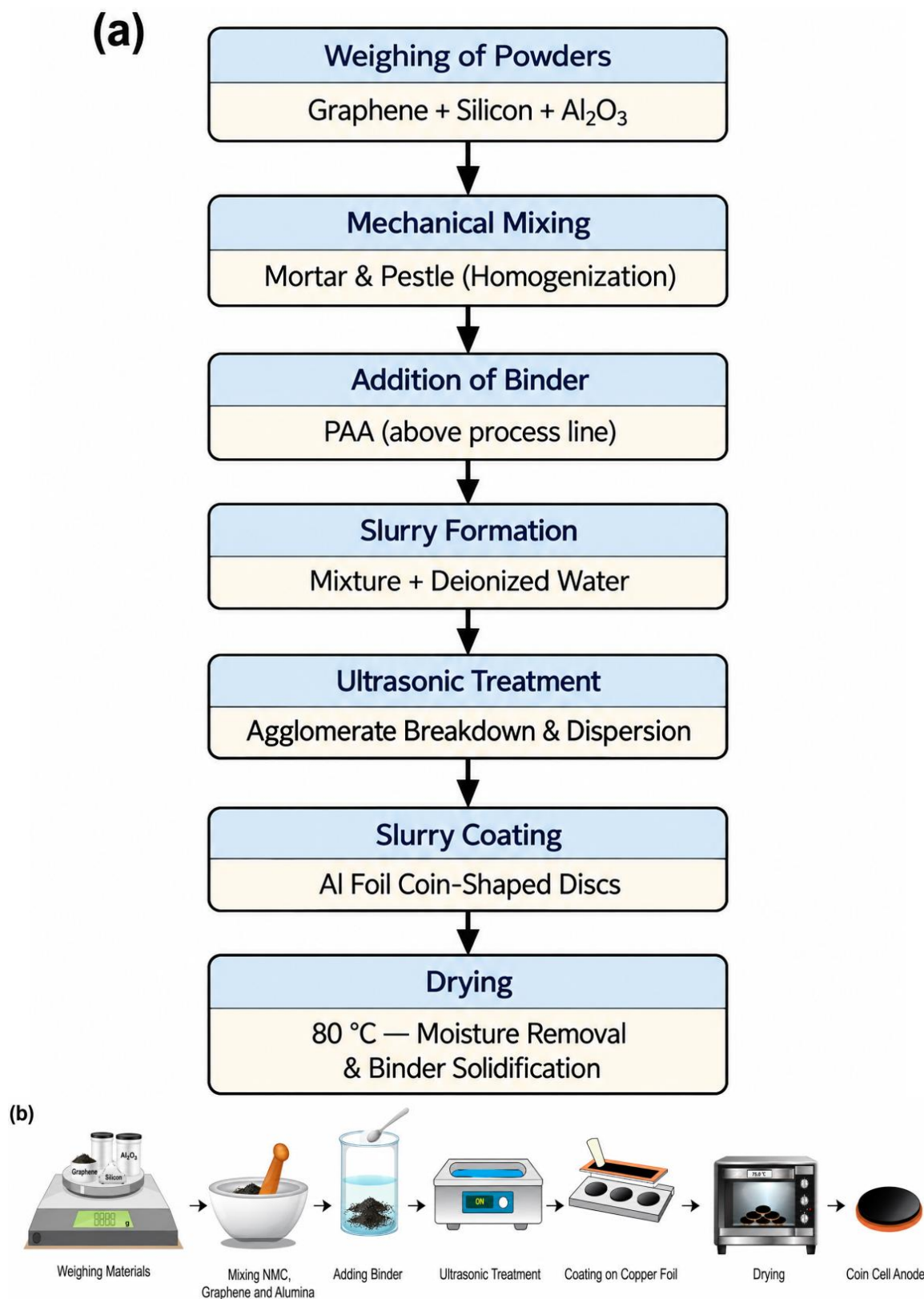


Figure 1. (a) Flow-chart and (b) Schematic of Si-graphene-Al₂O₃ composite anode fabrication.

Figure 1 shows the flow chart and schematic illustration of the fabrication process of Si-graphene-Al₂O₃ composite anodes. The purpose of this fabrication process was to obtain a homogeneous anode, where the individual components would be evenly distributed. Initially, the required amounts of silicon, graphene, and Al₂O₃ powders were carefully weighed according to the desired composition and each sample was thoroughly mixed using a mortar and pestle to achieve a homogeneous blend. This kind of mixing was particularly important for achieving a uniform

distribution of the graphene and Al_2O_3 within the silicon matrix, which can contribute to improved electrical connectivity and structural stability of the composite electrode. After the mixing process, polyacrylic acid (PAA) was added as a polymeric binder to improve adhesion and mechanical stability. The prepared powder-binder mixture was then transferred to a beaker with an appropriate amount of deionized (DI) water to form an aqueous electrode slurry. Subsequently, the slurry was subjected to ultrasonic treatment using an ultrasonic cleaner. The sonication process ensured a fine, uniform dispersion of nanoparticles by breaking agglomerates and enhancing interfacial bonding among graphene, silicon, and Al_2O_3 particles. After ultrasonic treatment, the resulting homogeneous slurry was evenly coated onto copper foil substrates using a thin-film spreader to maintain uniform thickness. The coated foils were then dried at 80°C to remove moisture and solidify the binder network. Finally, the composite electrodes were dried and small coin-shaped disks with the desired size were cut out for testing.

2.3. Surface Morphology Characterization

Scanning electron microscopy is a widely used characterization technique for examining the surface morphology, structural features, and microstructure of fabricated composite materials. In this study, the surface morphology of the fabricated anodes was examined using a scanning electron microscope (Hitachi SU1510) equipped with a vacuum system. For SEM characterization, solid specimens measuring approximately $2\text{ mm} \times 2\text{ mm}$ were carefully cut from the fabricated anodes to obtain representative samples while minimizing surface damage. The specimens were adhered to a conductive copper sample plate using conductive carbon tape before imaging. The carbon tape provided mechanical bonding between the samples and the sample holder and ensured electrical conductivity during SEM analysis. The specimens were then coated with a thin layer of platinum (Pt) and imaged.

2.4. Chemical Functional Group Analysis

The chemical functional groups of the fabricated composite materials are usually identified using FTIR. FTIR is also well known for identifying the molecular bonding present in fabricated composite materials. In this study, FTIR analysis was performed to investigate the chemical structure and functional groups present in the fabricated anode samples. A Thermo Scientific Nicolet iS5 FTIR spectrometer was employed to obtain the FTIR spectra of the fabricated silicon-graphene-aluminum oxide ($\text{Si-G-Al}_2\text{O}_3$) composite samples. For the analysis, a thin film was prepared and evenly deposited onto clean glass plates. The prepared specimens were then placed in the FTIR spectrometer for spectral acquisition. The measurements were conducted over a wavenumber range of $450\text{--}4000\text{ cm}^{-1}$, which covers the characteristic vibrational regions associated with a wide range of inorganic and organic functional groups.

2.5. Phase Structure Analysis

The X-ray diffraction (XRD) analysis is a characterization technique widely used to observe the crystallinity, structural properties, phase composition and crystalline structure of fabricated composite materials. XRD was employed to characterize the fabricated silicon-graphene-aluminum oxide ($\text{Si-G-Al}_2\text{O}_3$) composite anodes and to identify the crystalline phases present within the composite, where the diffraction intensities were recorded over a range of diffraction angles (2θ). For this purpose, a Bruker D8 Advance (or D8 Discover) (Manufacturer: Bruker AXS GmbH) device was used to investigate the structural properties of the composite anodes at room temperature. During the measurement, the intensity of the diffracted X-rays was collected at a scanning rate of $3^\circ/\text{min}$ with a step interval of 0.05° over the 2θ range of $10\text{--}80^\circ$. This range and step size are sufficient to provide better angular coverage and resolution to identify the diffraction peaks of the corresponding individual materials.

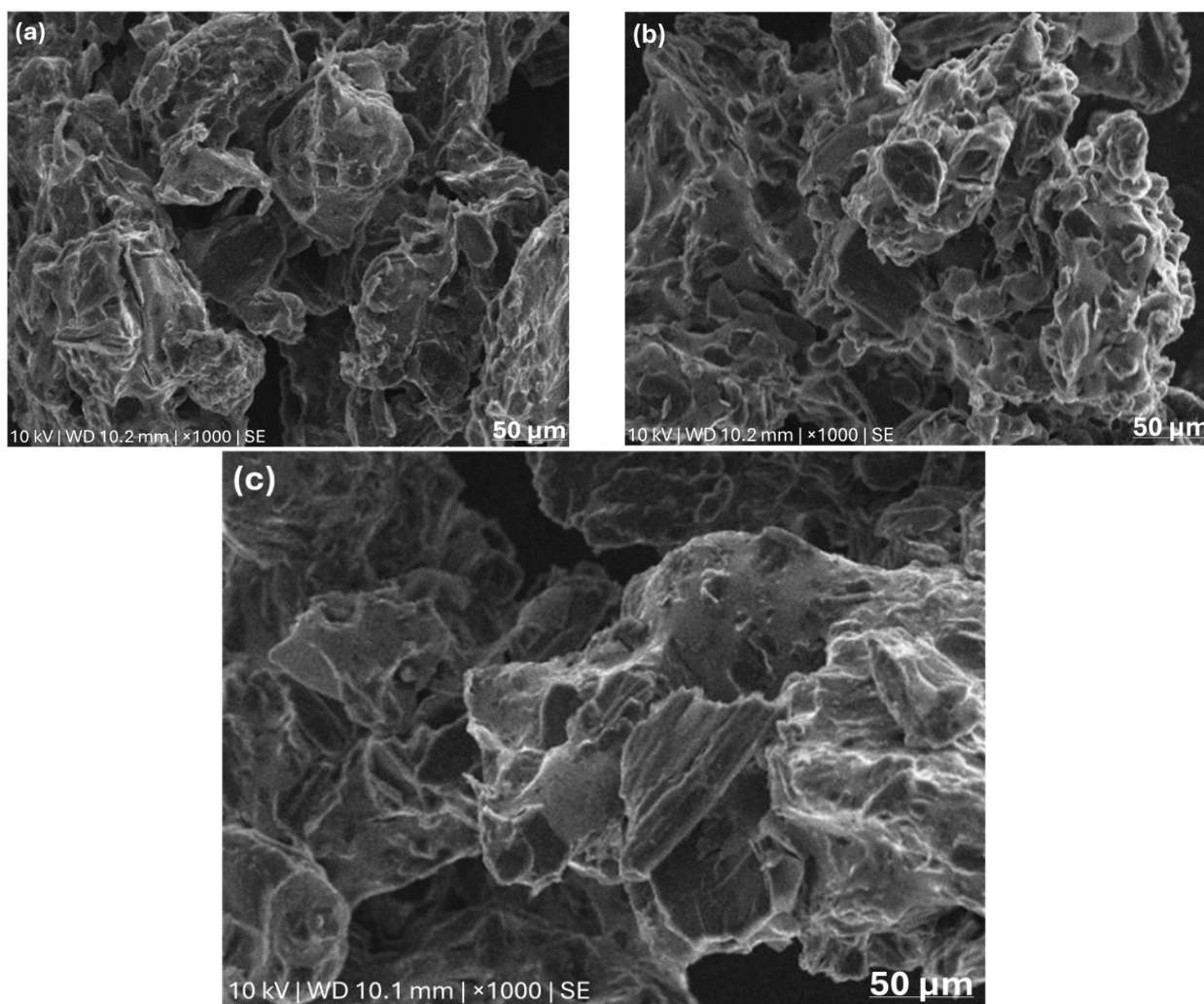


Figure 2. SEM micrographs of the fabricated silicon–graphene–aluminum oxide (Si–G–Al₂O₃) anode samples: (a) Sample 1 (S1), (b) Sample 2 (S2), and (c) Sample 3 (S3).

3. RESULTS AND DISCUSSION

3.1. Surface Morphology Characterization of the Fabricated Anode

Figure 2 shows the SEM micrographs of the fabricated silicon–graphene–aluminum oxide (Si–G–Al₂O₃) anode samples. These images illustrate the overall surface morphology, particles and size distribution. All the composite samples exhibit rough and porous surfaces with loosely packed particles, and the wrinkled, crumpled structures form agglomerates, characteristic of graphene-based materials. All the micrographs reveal a rough, porous surface texture, suggesting effective nanoparticle anchoring. These features are very common in graphene-based materials. The porous morphology of the anode samples is mainly covered with graphene and silicon particles. The graphene sheets form irregular agglomerates around silicon particles. This structure provides physical contact between the active material and the conductive graphene network. The silicon particles are distributed over and between the graphene sheets. The Al₂O₃ nanoparticles are not clearly distinguishable in the SEM images. This may be due to their small size and relatively uniform distribution throughout the composite. This highly porous electrode surface can enhance lithium-ion and electrolyte transport by facilitating electrolyte penetration, improving electrical conductivity, and accommodating silicon volume expansion during oxidation-reduction.

The morphology of Sample 1 is highly porous with a high concentration of graphene in Figure 2(a). There is a limited amount of silicon on the surface. The graphene sheets are organized into large, irregular sheets where silicon only covers a small portion. The percentage of available active material for Lithium storage may be diminished due to the low silicon concentration. As a result, the overall charge-storage capacity of Sample 1 might be lower.

The morphology of Sample 2 shown in Figure 2(b) is more compact and uniform. The smaller ones are spread around the surface. A greater amount of silicon is also observed compared with Sample 1. The particles of silicon are well linked to the graphene network. This structure is favorable for providing better contact between the active material and conductive phase. It can also help to minimize the development of isolated silicon regions during cycling. The surface is relatively uniform, allowing for more uniform lithiation and delithiation.

Sample 3 in Figure 2(c) shows an intermediate morphology between Samples 1 and 2. Silicon particles are seen attached to several graphene flakes. The distribution of silicon, however, is not as even as in Sample 2. There is some localized particle aggregation. Small gaps are also visible between the particles. These gaps can allow electrolytes to get in. But with excessive aggregation, contact between silicon and graphene could be diminished and can cause local mechanical instability during cycling.

From the SEM results, it is found that there is a noticeable effect of silicon content on the composite morphology. The structure of sample 1 is more open and graphene-like. Sample 2 has a more even and concentrated distribution of silicon particles. Sample 3 has intermediate properties, with some silicon aggregation present at local scales. The three samples have different ratios of silicon particles and the graphene network, with the ratio being the most uniform in Sample 2. This morphology can offer good electron and lithium-ion conducting channels. It can also provide more structural strength during repeated lithiation-delithiation. Based on the SEM analysis, Sample 2 is found to have a favorable microstructure that will enable the production of better anode performance and cycling stability.

3.2. Chemical Functional Group Analysis of the Fabricated Anode

Using FTIR, the samples were scanned to identify functional groups and chemical bonds in the fabricated silicon-graphene-aluminum oxide (Si-G-Al₂O₃) anodes. The spectra of all fabricated anode samples exhibited multiple transmittance peaks in the wavenumber range of 4000-400 cm⁻¹. It is important to note that the FTIR spectra of the synthesized Si-G-Al₂O₃ composites were very similar overall, except for slight fluctuations in wavenumbers and transmittance, indicating that the sample compositions were the same across samples, except for the variations in weight percentage. The composite matrix had vibrations of C-C, C=C, Si-O, Al-O, and O-H.

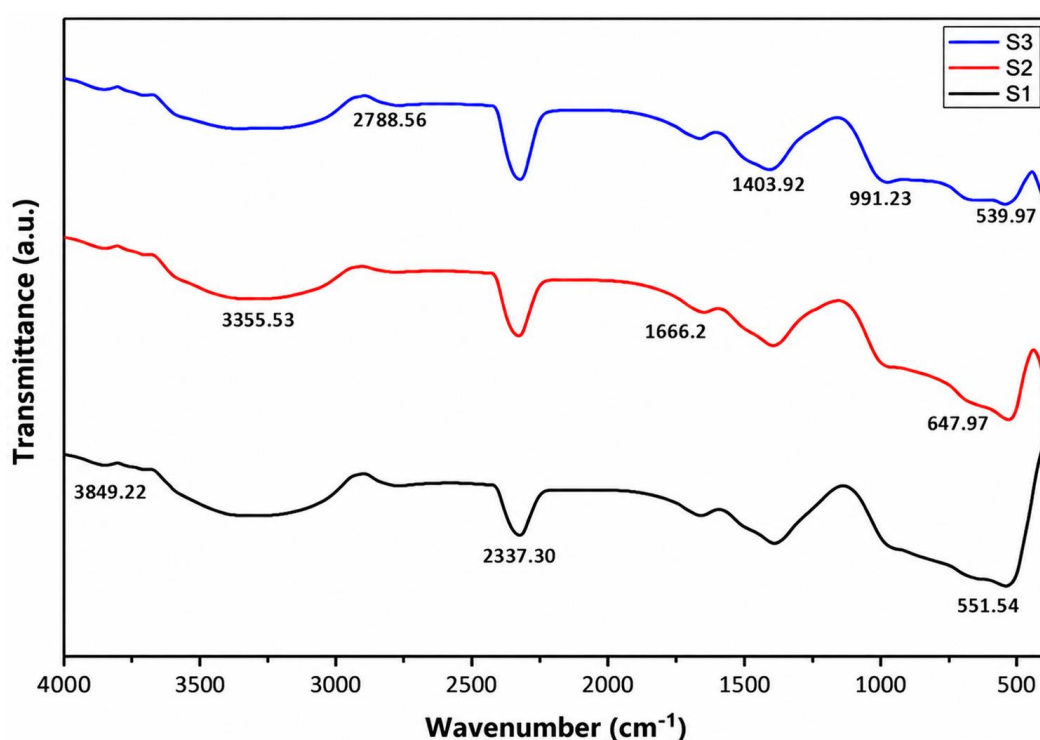


Figure 3. FTIR spectra of the fabricated silicon-graphene-aluminum oxide (Si-G-Al₂O₃) anode samples.

As shown in the FTIR transmittance (%) spectra in Figure 3, a small band at $\sim 3849\text{ cm}^{-1}$ is attributed to free-surface O–H stretching vibrations, possibly due to the isolation of hydroxyl groups on alumina. In the FTIR spectrum of the Si–Gr–Al₂O₃ composite, some absorption bands are observed corresponding to the surface functional groups and chemical bonds. It is seen that the absorption band is very broad in the range $3660\text{--}2900\text{ cm}^{-1}$. The main association of this band is with O–H stretching vibrations of surface hydroxyl groups and adsorbed moisture. This wide band indicates the occurrence of hydroxyl groups on the composite surface that are capable of hydrogen bonding. A weak absorption band is found near 2788 cm^{-1} . This is a band that can be correlated with aliphatic C–H stretching vibrations. It could come from the organic binder or trace quantities of organic species that are left over from the process of making the electrodes. It is also an indication of the retention of some carbon-containing surface groups in the composite. A well-defined peak is seen around 2337 cm^{-1} . It is the asymmetric stretching vibration of CO₂ that is associated with this peak. It can occur with the uptake of CO₂ from the atmosphere on the sample surface during handling or FTIR measurement. Thus, it should not be straightforwardly concluded that this peak is due to Si–H bonding. The bands of absorption around $1662\text{--}1660\text{ cm}^{-1}$ may be related to C=C stretching vibrations and surface-related O–H bending. The bands in the range of $1396\text{--}1415\text{ cm}^{-1}$ can be related to C–C skeletal vibrations and other surface functional groups. The peaks represent structural features inside the Si–Gr–Al₂O₃ composite that contain carbon. The characteristic C=C stretching vibration of graphene confirms the preservation of the sp² carbon framework (Ferrari et al., 2006).

A distinct absorption peak appears in the range of $985\text{--}992\text{ cm}^{-1}$. This peak can be assigned to Si–O–Si stretching vibrations. It indicates the presence of oxidized silicon species on the particle surface. The formation of a thin oxide layer may occur due to exposure of silicon to air during processing and handling. Additional absorption bands are observed near 647 cm^{-1} and in the range of $547\text{--}539\text{ cm}^{-1}$. These bands are associated with metal–oxygen and silicon–oxygen vibrations. The band near 647 cm^{-1} can be related to Al–O vibrations, while the lower-frequency bands may arise from Si–O and Al–O lattice vibrations. The strong absorption in this low-wavenumber region is consistent with the presence of oxide and alumina phases in the composite (Jella et al., 2023).

Overall, the FTIR bands in the lower-wavenumber region provide further evidence of Si–O and Al–O bonding within the Si–Gr–Al₂O₃ composite. These vibrations are consistent with the presence of the silicon oxide layer and Al₂O₃ phase in the fabricated electrode.

3.3. Phase Structure Analysis of the Fabricated Anode

X-ray Diffraction (XRD) was used to identify the phases present and the degree of crystallinity of the fabricated silicon–graphene–aluminum oxide (Si–G–Al₂O₃) composite anodes. Cu K α radiation was used to record the diffraction intensities of all the Si–G–Al₂O₃ anodes over a 2θ range of 10° to 80° .

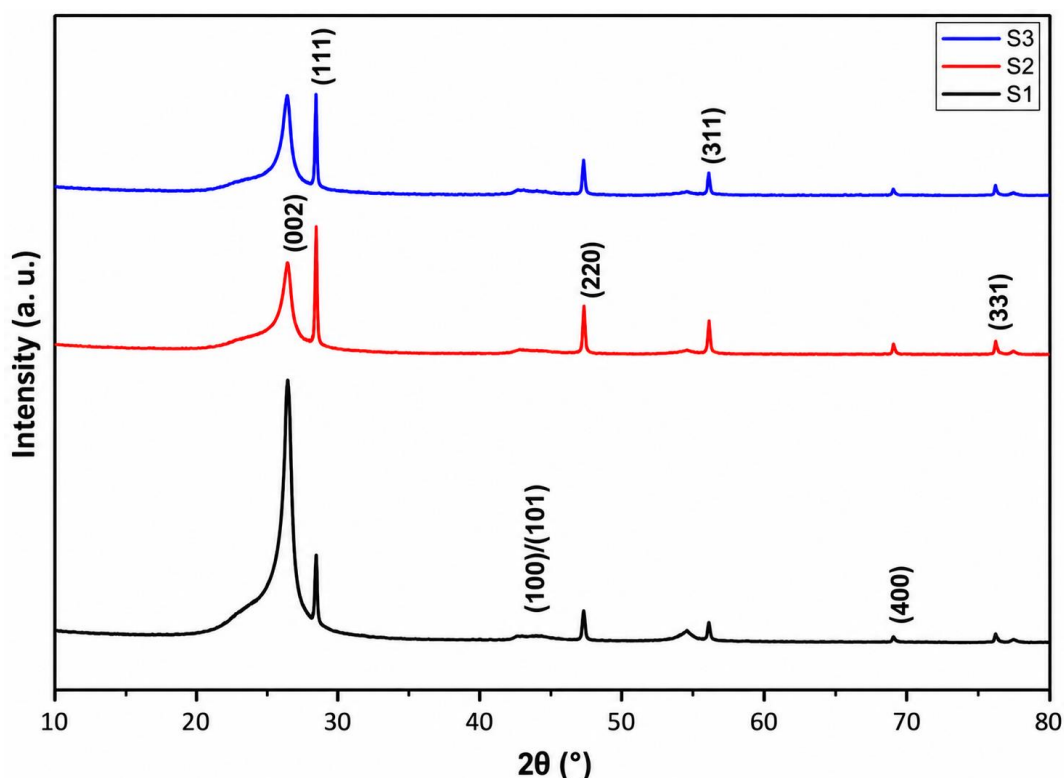


Figure 4. XRD spectra of the fabricated silicon-graphene-aluminum oxide (Si-G-Al₂O₃) anode samples.

The X-ray Diffraction (XRD) patterns of three samples of Si-G-Al₂O₃ composite materials are presented in Figure 4. The diffraction pattern is very similar for all three samples. But it is possible to see how the peak position and intensity have changed slightly. The changes might be attributed to the relative weight ratio of Si, graphene and Al₂O₃ in the fabricated samples. The patterns are also very similar, indicating the same main phases in all three samples.

The majority of the diffraction peaks are within the range of 25°–80°2θ. The intensity and form of an XRD peak can give information regarding the structure of the material. The sharp and intense peaks are typically associated with highly crystalline phases. On the other hand, the broad and weak peaks are typically indicative of low-crystallinity materials or extremely small crystallites.

In all the Si-G-Al₂O₃ composite samples, a broad diffraction peak is found around 2θ ≈ 26.20–26.35°. This peak can be attributed to the (002) lattice plane of graphene. The wide peak indicates that the graphene layers are not many and not well stacked. This observation indicates the presence of few-layer or partially exfoliated graphene in the composite. Additionally, the graphene phase can promote the development of a conductive network in the composite structure.

There are also a few sharp peaks in the XRD patterns. The peaks located at approximately 2θ ≈ 28.30–28.40°, 47.20–47.30°, and 54.02–56.05° can be attributed to the (111), (220), and (311) planes of crystalline silicon, respectively. The peaks are sharp, which shows the crystalline structure of the silicon phase. These slight differences in position and intensity of the samples may be due to compositional differences and interactions between the various components during composite preparation (Dash et al., 2022).

Additionally, a noticeable change in the peak shape can be observed with a decrease in the silicon content. The diffraction peaks are relatively sharp in the samples with low silicon content. This transformation could be a consequence of the distribution of silicon particles in the composite as well as the interaction between silicon, graphene and Al₂O₃. The sharpness of the peaks also reflects the change in crystallinity and microstructure of the composite.

The relative intensity and the peak broadening of the diffraction peak near $2\theta \approx 54.5^\circ$ vary slightly among the samples prepared. This peak is mainly associated with the crystalline silicon phase. Such changes in its intensity and width can be of great help in gaining information about changes in the microstructure inside the composite. The change in peak intensity may be associated with differences in the content and direction of the Si phase in the crystal. Likewise, a decrease in crystallite size, lattice strain, or changes in the local structure of the material may correspond to peak broadening. If a variation of this peak is observed, it indicates that the silicon structure is altered with the variation of the composite composition.

Besides the main silicon peaks, small diffraction peaks can be seen around $2\theta \approx 69.02 - 69.23^\circ$ and $76.25 - 77.65^\circ$. These peaks can be assigned to the (331) and (400) planes of crystalline silicon, respectively. These reflections further verify the crystallinity of the silicon phase in the prepared composites. These peaks are not as strong as the Si peaks, but they also indicate the presence of crystalline silicon.

There is also a broad and weak feature around $2\theta \approx 42.20 - 42.50^\circ$. This feature is possibly associated with the (100)/(101) planes of the carbon-based phase. The broadness of this feature indicates a relatively low degree of crystallinity and a disordered carbon structure. As the proportion of graphene in the composite is relatively small, the diffraction peaks of graphene are expected to be comparatively weak compared to the peaks of Si. Moreover, the characteristic diffraction features can be further broadened and weakened due to the few-layer character and partial exfoliation of graphene.

Notably, the XRD pattern of the anode samples doesn't show diffraction peaks of Al_2O_3 and other impurities, probably due to its low content, nanocrystalline or amorphous nature, and presence as an ultrathin interfacial coating on silicon and graphene. Additionally, Al_2O_3 may overlap or be composed of significant Si proportions, making it indistinguishable (Zhao et al., 2023).

The XRD results indicate the formation of Si-G- Al_2O_3 composite, with the predominant phase being crystalline silicon. The wide peak around 26° is due to graphene, and the sharp peaks are predominantly from crystalline silicon. The differences in peak intensity, position and broadening suggest differences in composition and microstructure between the samples. The weak and broad C-related features agree with the low graphene content and few-layer structure. There are no distinct peaks showing the presence of Al_2O_3 , which may be attributed to the presence of a low concentration of Al_2O_3 , an amorphous or nanocrystalline form, ultrathin distribution or overlap with the stronger Si peaks. The overall crystal structure of the samples is similar, and it is evident that the microstructural properties of the samples vary with their Si, graphene, and Al_2O_3 content, which may influence their electrochemical properties.

4. CONCLUSIONS

Despite the significant progress in lithium-ion batteries, conventional anode materials still face limitations such as limited specific capacity, poor rate capability, structural degradation, and volume expansion during repeated cycling. Composite anodes have emerged as a promising strategy to combine the complementary properties of different materials, enabling improved electrical conductivity, structural stability, cycling performance, and overall energy-storage capability, as composite materials are well known for combining the properties of the individual constituent materials in a favorable manner. Graphene has been used as a promising component material for composite anodes because of its excellent electrical conductivity, high surface area and superior mechanical properties. The properties of graphene make it especially promising as an anode component material and as a conductive matrix to improve the electrochemical properties and structural stability of composite anodes. In this article, silicon-graphene-aluminum oxide (Si-G- Al_2O_3) composite anodes were synthesized and characterized as potential high-performance anodes for lithium-ion batteries. Silicon was incorporated for its high theoretical energy capacity (~ 3200 mAh/g), whereas graphene provides excellent electrical conductivity and flexibility, and Al_2O_3 maintains mechanical and chemical stability. Three anode samples with different compositions, namely S1 (94% G, 5% Si, 1% Al_2O_3), S2 (84% G, 15% Si, 1% Al_2O_3), and S3 (89% G, 10% Si, 1% Al_2O_3), were synthesized by mortar and pestle for a

homogeneous mixture. Polyacrylic acid (PAA) binder was then added and sonicated in deionized water to provide uniform nanoparticle dispersion, break up agglomerates, and enhance interparticle bonding. A variety of analytical methods, including SEM, FTIR, and XRD, were employed to confirm the successful synthesis of chemically and structurally stable Si–G–Al₂O₃ anodes. Characterization images showed that, among the samples, sample-2 (84% G, 15% Si, 1% Al₂O₃) displayed the finest morphology and mechanical integrity owing to its composition and may have potential for high-performance lithium-ion batteries.

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Transparency: The authors state that the manuscript is honest, truthful, and transparent, that no key aspects of the investigation have been omitted, and that any differences from the study as planned have been clarified. This study followed all writing ethics.

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Authors' Contributions: All authors contributed equally to the conception and design of the study. All authors have read and agreed to the published version of the manuscript.

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