



Green synthesis and characterization of Graphene/SnO₂ nanocomposite photoanodes for enhanced DSSC performance

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Abstract

Dye-sensitized solar cells (DSSCs) offer a compelling alternative to conventional silicon-based solar cells due to their cost-effectiveness, flexibility, and relatively high efficiency. However, their performance is currently hindered by the photoanode material, typically titanium dioxide (TiO₂). This study aims to develop a green synthesis method for graphene/tin dioxide nanocomposites (G/SnO₂ NCs) using *Bryophyllum pinnatum* extract for DSSC applications. SnO₂ nanoparticles (SnO₂ NPs) were synthesized using a green method with *Bryophyllum pinnatum* extract and integrated with biomass-derived graphene to fabricate G/SnO₂ NCs for DSSC applications. Characterization techniques, including UV-Vis spectroscopy, XRD, FTIR, SEM, and EDX, were employed to analyze the optical, structural, functional, morphological, and elemental compositional properties of graphene, SnO₂, and the G/SnO₂ NCs. The photoanode thin films were deposited using the doctor blade technique, and their electrical properties were evaluated using four-point probe measurements. Results demonstrate that the G/SnO₂ NCs exhibit significantly enhanced electrical conductivity (0.148 S/m) compared to pristine SnO₂ (0.098 S/m) and graphene (0.122 S/m), indicating improved charge transport properties within the composite material. This enhancement is attributed to the synergistic effect of the high electron mobility of SnO₂ and the excellent conductivity of graphene. Furthermore, the G/SnO₂ NCs exhibit lower sheet resistance (549.48 Ω), further suggesting its potential for efficient charge collection in DSSC applications. The graphene/SnO₂ nanocomposites exhibited enhanced electrical conductivity, improved charge transport properties, and lower sheet resistance compared to pristine SnO₂ and graphene. These findings suggest that the synergistic combination of SnO₂ and graphene offers a promising pathway to improved efficiency in DSSC applications. This research contributes to the development of sustainable and cost-effective solar energy solutions, offering a promising alternative to conventional silicon-based solar cells.

1. Introduction

Renewable energy provides a sustainable and eco-friendly alternative to fossil fuels, lessening oil dependence, cutting greenhouse gas emissions, and combating the climate crisis [1–3]. Renewable energy comes from sunlight, either directly or indirectly, through

methods like heating the earth's surface and the types include solar, wind, hydropower, biomass, and geothermal [4]. Solar energy, an affordable and clean power source, drives wind, water, bio-energy, and fossil fuels. Solar cells, typically silicon-based, convert sunlight to electricity with 15-20%

efficiency [5]. High costs and harmful chemicals in silicon production push researchers to explore eco-friendly alternatives like dye-sensitized solar cells (DSSCs), which are cheaper to manufacture [6,7]. DSSCs convert solar energy to electricity using wide-band semiconductor and dyes. They consist of a counter electrode, electrolyte, sensitizer, and a porous semiconductor electrode. The dye absorbs and converts light into electrical energy [8]. Typical DSSC photoanodes consist of a transparent conducting oxide (TCO) substrate, wide-band oxide semiconductor, and dye sensitizer.

Titanium dioxide (TiO_2) is commonly used for its beneficial properties but has low carrier mobility, leading to research into alternatives like Tin dioxide (SnO_2), an n-type semiconductor with high electron mobility [9,10]. Despite advancements, SnO_2 -based DSSCs still struggle with poor dye adsorption and low open circuit voltage (Voc) [11]. Enhancing electron transport in SnO_2 can boost DSSC efficiency. Graphene, with its excellent conductivity, can significantly improve electron transport [12] and reduces charge recombination in photoanodes, enhancing photovoltaic cell efficiency [13]. Graphene-based materials provide high charge mobility, increased surface area, and excellent electrical conductivity.

Green synthesis is crucial for minimizing hazardous chemicals, reducing energy consumption, lowering costs, and promoting sustainability by cutting waste and pollution [14]. In bottom-up synthesis, nanoparticles form from atoms and molecules through reduction/oxidation resulting in minimal defects and uniform composition [15–17]. Biological methods often use microorganisms and plant extracts to synthesize nanoparticles [18–20].

Green synthesis of hybrid structures using controllable SnO_2 and graphene boosts electron transfer and dye adsorption, improving DSSC performance. However,

DSSCs performance is often hindered by photoanode materials like SnO_2 [21]. Despite its wide band gap and chemical stability, SnO_2 suffers from low electron mobility and poor charge transport efficiency [22]. Researchers have explored the incorporation of graphene due to its high electron mobility and excellent conductivity into the SnO_2 NPs [23]. This integration enhances electron transport properties and accelerates charge carrier mobility, thereby improving DSSC efficiency [24]. Therefore, this research explores the ways in which graphene enhances the performance of SnO_2 -based photoanodes in DSSCs, focusing on the improvement of electron mobility and charge transport efficiency.

2. Materials and Methods

2.1 Materials

Bryophyllum Pinnatum shoot and leaves were harvested in the nearest farm garden in Ogbomoso. All chemicals, solvents, and reagents were used as supplied, without additional purification. SnCl_2 (99% pure) was purchased from Sigma-Aldrich, and deionized (DI) water was used in all solutions throughout the study.

2.2 Synthesis of Graphene using *Bryophyllum Pinnatum* shoot

The plant shoot, sourced from a local farm in Ogbomoso, Oyo State, Nigeria, was chosen for biosynthesis due to its reducing properties. The plant shoot was washed with deionized (DI) water to remove impurities, then air-dried and ground into a fine powder. The powder was carbonized in an electric furnace at 200 °C for 2 hours and stored for future use. A 3 g sample of the carbonized *Bryophyllum Pinnatum* shoot plant was mixed with 250mL of 2.5M HNO_3 (2.50 mol L^{-1}) and shaken at 300rpm at 70 °C for 300 minutes. The mixture was filtered 20 times through a 0.45 μm glass fiber filter paper. The resulting black precipitate was separated by centrifugation at 3000rpm for 10 minutes. The residue was washed three times with

ethanol to remove impurities and dried at 120 °C for 2 hours. The final product was then collected [25].

2.3 Preparation of *Bryophyllum Pinnatum* leaves Extract

The *Bryophyllum Pinnatum* leaves were washed thoroughly with distilled water to remove the dust particles and dried under sunlight. 10 g of dried leaves were subsequently grinded to obtain fine powder and dissolved in 100 ml DI water. The suspension was boiled for 4 h at 60 °C and cooled down to room temperature. Eventually, the solution was filtered by Whatman No. 1 filter paper to obtain brown coloured leaf extract. The extract was then cooled down and stored in the refrigerator (4° C) for utilization in the synthesis of SnO₂ NPs.

2.4 Preparation of Tin dioxide Nanoparticles (SnO₂ NPs)

Tin chloride (SnCl₂) of 3.5 g was solubilized in 40 ml DI water and was stirred for 15 minutes. Subsequently 20 ml of prepared *Bryophyllum Pinnatum* leaf extract was added to the SnCl₂ solution, followed by heating at 60 °C for 4 h. Then resultant mixture was kept on hot plate until jelly was formed. The jelly was finally calcinated at 400 °C for 4 h in electric furnace to obtain dark brown powder of SnO₂ NPs [26].

2.5 Preparation of Graphene/Tin dioxide Nanocomposite

G/SnO₂ nanocomposite with different concentrations of graphene (G) was synthesized using the Mixing solution method. The prepared SnO₂ NPs and G and ethanol were used as the starting materials for the synthesis of the G/SnO₂ nanocomposite. Firstly, 10 mg of the obtained G powder was dispersed in 10 mL of ethanol and 200 mg of SnO₂ was dispersed in 20 mL of ethanol. These two solutions were then mixed homogeneously using an ultrasonic bath for 1 hour at room temperature and then transferred into a 100 mL treated at 180 °C for 24 hrs. The final products were centrifuged, washed with ethanol and distilled water, and then dried at 60 °C for 12 hrs to obtain G/SnO₂ nanocomposite [27].

2.6 Film deposition of Graphene (G), SnO₂ NPs and G/SnO₂ NPs) using doctor-blade method

The synthesized SnO₂ and G/SnO₂ nanocomposite were made into a paste by mixing 0.5g of G/SnO₂ nanocomposite powder with 10 ml of distilled water and in a mortar for about 30 min using 0.5g of starch as a dispersing agent then poured into a clean beaker. Distilled water (10 ml) was added to break the aggregates into separate particle; the mixture was stirred for 30 minutes at room temperature. Then, ethanol (10 ml) was added at intervals and the mixture was stirred for 30 minutes to obtain a homogeneous

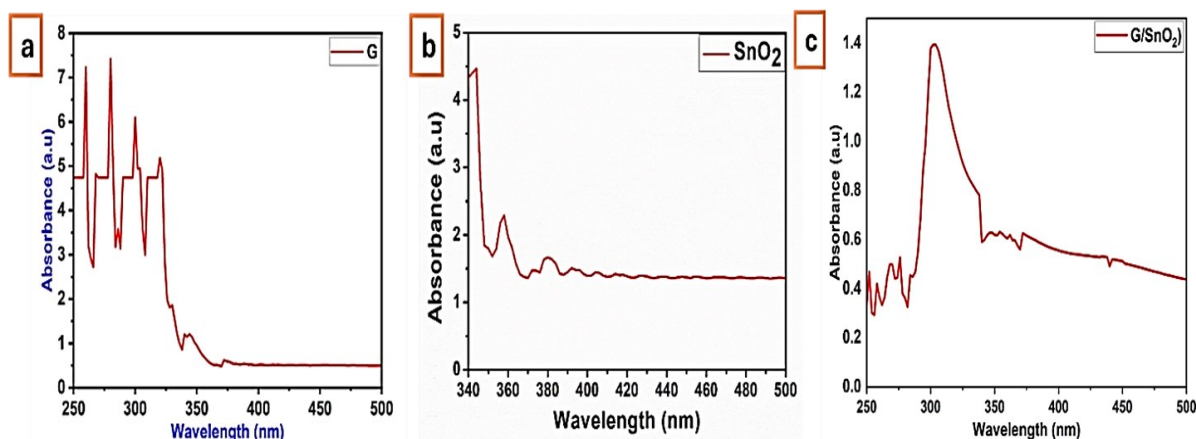


Fig. 1: UV-VIS absorption spectra of synthesized (a) G (b) SnO₂ NPs and (c) G/SnO₂ NCs

paste. The pastes (1.0 ml each) were incorporated, the doctor blade method used for applying pastes in all cases and the area of the applied layer was 2.0 x 2.0 cm [28–30]. The process was repeated for both Graphene and SnO₂ NPs respectively, the edge of 0.5 cm by 0.5 cm was made using adhesive tape on a conducting side of the FTO such that FTO was first covered on two parallel edges with adhesive tape to control the areas of the films and to provide non-coated areas for electrical contact. The obtained pastes were applied to one of the free edges of the conducting glass substrates and distributed with a glass rod (spatula) sliding over the tape-covered edges. Also, the reference electrode was prepared without nanoparticles. They were left for 20 minutes to relax to reduce the surface irregularity and then dried for 45 mins at 60 °C. Allow the film to dry for one hour until it is completely solidified. Perform additional post-processing steps such as annealing or sintering to improve the film's properties [31].

2.7 Characterization

The analysis for the crystalline structure, optical properties, surface morphology, and molecular compositions were carried out. The UV-Vis absorption of extracts and nanoparticles was measured using a double beam UV-Vis-IR spectrophotometer (ASUV-6300PC) with a Xe lamp, covering wavelengths from 200 - 800 nm. Aqueous samples were injected into a quartz cuvette for the UV

-Vis experiment. X-ray diffraction (XRD) analysis was used for phase identification of graphene (G), tin dioxide (SnO₂) nanoparticles, and G/SnO₂ nanocomposites on the Rigaku D/Max-III C X-ray diffractometer. X-ray diffraction provides detailed crystallographic structure information and is standard for determining the atomic structure of crystalline materials. The average crystallite size (D) was calculated using the Scherrer equation as shown in equation 1[32]:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad \dots \dots \dots 1$$

FTIR studies of extracts and nanoparticles were performed using a NICOLET iS10 spectrometer, within the mid-infrared range (400-4000 cm⁻¹) at room temperature with KBr as a diluting agent. The morphology and composition of graphene (G), tin dioxide (SnO₂) nanoparticles, and Graphene (G) incorporated into SnO₂ were examined using SEM (JOEL-JSM 7600F). Energy Dispersive X-ray (EDX) Model INCA 200 (UK) at 20 keV, paired with SEM, analyzed elemental composition, percentage, and purity of synthesized metal oxide nanoparticles. The current-voltage (I–V) characteristic of synthesized samples have been examined using a Four-Point Probes (4 PP) system equipped with Keithley 2400 sources measuring unit.

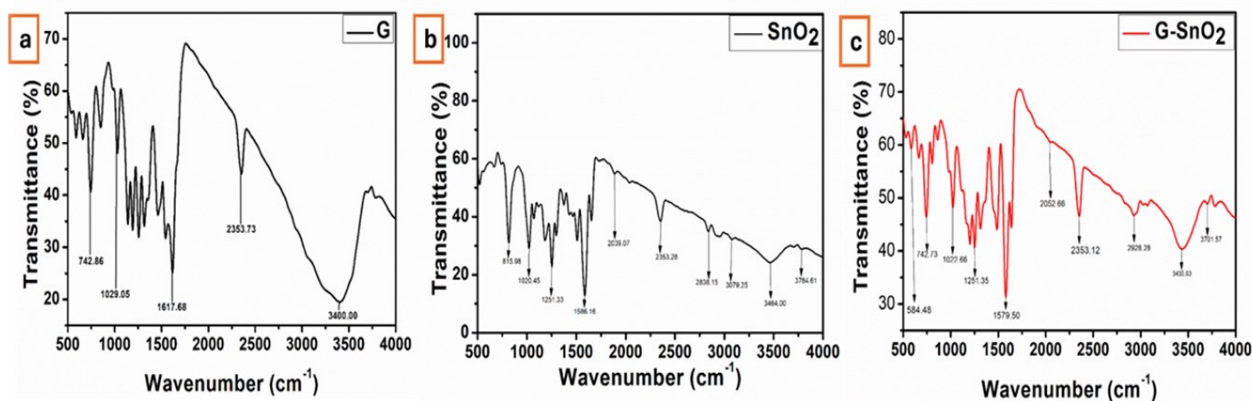


Fig. 2: FTIR of synthesized (a) G (b) SnO₂ NPs and (c) G/SnO₂ NCs.

Table 1: Summary of FTIR Analysis of G, SnO₂ NPs and G/SnO₂ NCs

Wavenumber Ranges (cm ⁻¹)	G	SnO ₂ NPs	G/SnO ₂ NCs	Functional Group	Bond Type
584.48 - 742.86		Sn-O stretch	Sn-O stretch	Sn-O stretch	Sn-O
1026.48 1170.12	- C-O stretch		C-O stretch	C-O stretch	Ester, Ether
1384.98 1617.68	- C=C stretch	C-H bend	C=C stretch	C-H bend, C=C stretch	Alkane, Alkene
1917.68	C=O stretch	C=O stretch		C=O stretch	Carbonyl
2363.73 2924.57	- C-H stretch	C-H stretch	C-H stretch	C-H stretch	Alkane
3206.37 3460.00	- O-H stretch	O-H stretch	O-H stretch	O-H stretch	Hydroxyl, Alcohol

3. Results and Discussion

3.1 Ultraviolet-visible (UV-Vis) Analysis of the synthesized samples

The UV-Vis absorbance spectrum of graphene (Figure 1(a)) ranges from 250-500 nm, peaking around 275 nm due to the π - π^* transition of C=C bonds. Absorbance sharply declines beyond 350 nm, indicating limited visible region absorption. SnO₂ NPs formation is first indicated by a colour change from colourless to pale yellow, then to dark brown, due to Sn (IV) reduction by *Bryophyllum Pinnatum* extract. The UV-Vis spectrum of tin dioxide (Figure 1(b)) peaks around 340 nm, decreasing and stabilizing beyond 400 nm, typical of tin oxide's UV absorption property [33].

The UV-Vis absorbance spectrum of G/SnO₂ NCs (Figure 1(c)) peaks around 275 nm, indicating graphene sheets, and around 350 nm, suggesting interaction with tin dioxide.

Overall absorbance is lower than individual components but covers visible light. These results corroborate well with synthesis of G/SnO₂ NCs reported by Khan and Parveen (2020) [34]. Graphene's incorporation into SnO₂ enhances optical properties, with improved absorption up to 372 nm, boosting conductivity and improve charge transport in DSSC.

3.2 Fourier Transform Infrared Spectrometry (FTIR) Spectroscopy of the synthesized samples

Functional groups on the nanoparticles' surface were identified using an FTIR spectrometer (NICOLET iS10). Figure 2 shows the spectra for Graphene (G), Tin dioxide Nanoparticles (SnO₂ NPs), and Graphene/Tin Dioxide Nanocomposite (G/SnO₂ NCs), while Table 1 lists wavenumber values and absorption characteristics. the

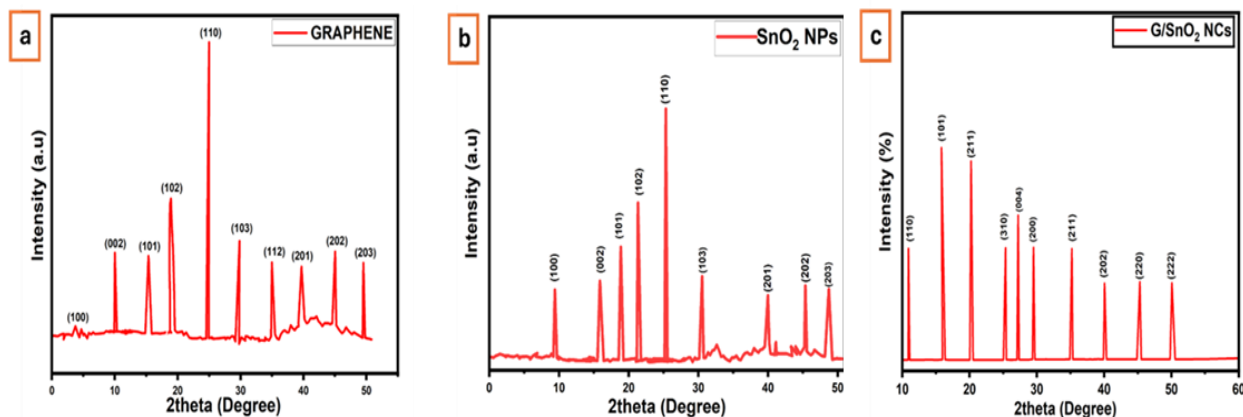


Fig. 3: XRD of synthesized (a) G (b) SnO₂ NPs and (c) G/SnO₂ NCs.

FTIR spectrum of synthesized G, shown in Figure 2(a), ranges from 500 to 4000 cm^{-1} . Notably, the hydroxyl compound ($-\text{OH}$) peak appears at 3400.00 cm^{-1} , and the C-H functional group peak is at 2353.73 cm^{-1} .

Furthermore, the FTIR spectrum identifies the aromatic carbon group ($\text{C}=\text{C}$) at 1617.68 cm^{-1} and the alkoxy group ($-\text{C}-\text{O}$) at 1029.05 cm^{-1} . A broad peak at 742.86 cm^{-1}

bending), and 1626.05 cm^{-1} ($\text{C}=\text{C}$ stretching). Peaks at 2363.73 cm^{-1} and 2924.57 cm^{-1} correspond to C-H stretching vibrations, and the broad peak around 3460.00 cm^{-1} represents O-H stretching vibrations, indicating the presence of hydroxyl group [33]. Figure 2(c) shows FTIR spectrum of the G/SnO₂ NCs shows main peaks that indicate the successful integration of G and SnO₂. The

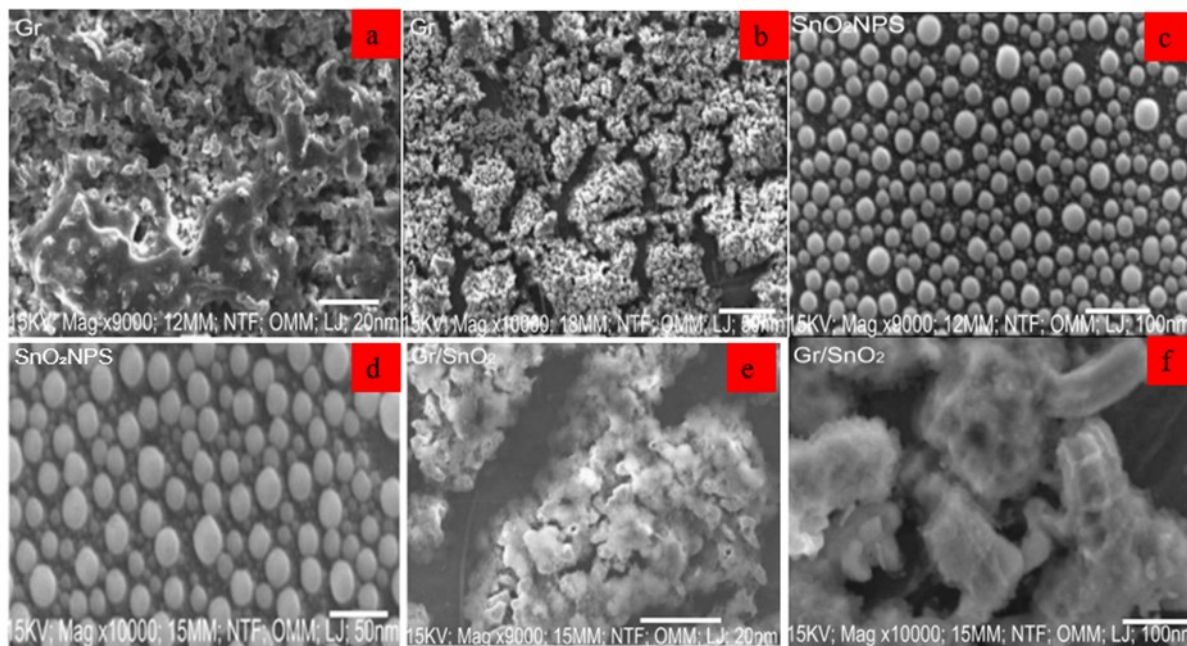


Fig. 4: SEM Micrograph of synthesized G (a and b), SnO₂ NPs (c and d) and G/SnO₂ (e and f)

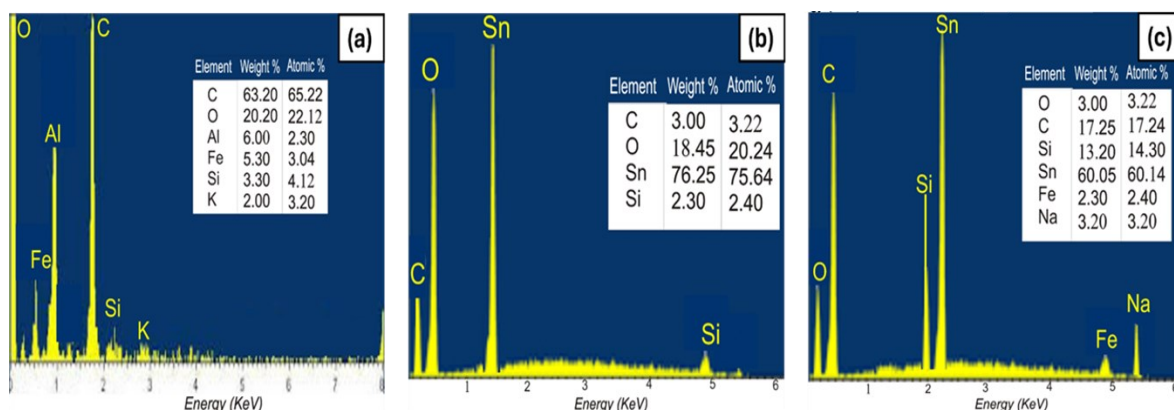


Fig. 5: EDX of synthesized (a) G (b) SnO₂ NPs and (c) G/SnO₂ NCs.

indicates $\text{C}=\text{O}$ vibrations, remnants of the carbonyl group during reduction [35] spectrum reveals typical graphene molecular vibrations. Figure 2(b) shows the FTIR spectrum of SnO₂ NPs, with peaks at 1026.48 cm^{-1} (Sn-O stretching), 1384.98 cm^{-1} (C-H

peak at 584.48 cm^{-1} represents Sn-O stretching vibrations. Peaks at 1170.12 cm^{-1} and 1384.98 cm^{-1} indicate C-O stretching and C-H bending vibrations, respectively. The peak at 1626.05 cm^{-1} represents $\text{C}=\text{C}$ stretching vibrations, while the peak at 1917.68 cm^{-1}

indicates C=O stretching vibrations. Peaks at 2363.73 cm^{-1} and 2924.57 cm^{-1} correspond to C-H stretching vibrations, and the broad peak around 3460.00 cm^{-1} represents O-H stretching vibrations, confirming the presence of hydroxyl groups [31].

3.3 X-ray diffraction (XRD) analysis of the synthesized samples

The XRD pattern for graphene shows prominent peaks at specific 2θ values (Figure 3(a)), indicating its crystalline structure and high crystallinity. The main peaks are labelled with their Miller indices: (100), (002), (101), (102), (110), (103), (112), (201), (202), and (203). Figure 3(b) shows the XRD pattern for SnO_2 nanoparticles with distinct peaks corresponding to the tetragonal rutile structure, labelled with their Miller indices: (100), (002), (101), (110), (103), (200), (211), and (220), confirming their crystalline nature and phase purity. The XRD pattern for the G/ SnO_2 nanocomposite (Figure 3(c)) displays peaks for both graphene and SnO_2 , indicating successful integration. Characteristic peaks include (002) for graphene, and (110), (101), (200), (211), (220), (002), (310), (112), (301),

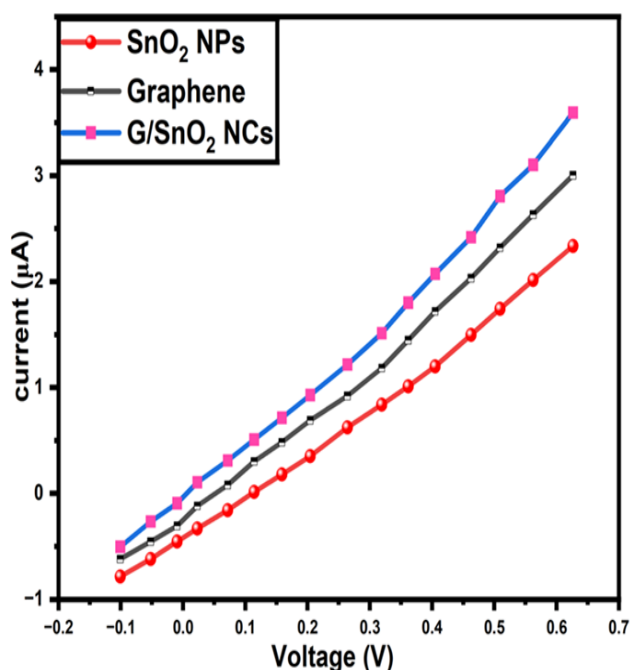


Fig. 6: Graph of Current versus Voltage for G, SnO_2 NPs, and G/ SnO_2 NC Thin Films

(202), (321), and (222) for SnO_2 , confirming the composite's composition and enhanced properties.

3.4 Scanning Electron Microscopy (SEM) of the Samples

In Figure 4(a), the 9000x magnification and 20 nm scale bar reveal graphene's rough, textured surface with small protrusions and depressions, indicating its nanoscale structure from defects, impurities, or functionalization. Figure 4(b), at 10000x magnification with a 50 nm scale bar, shows a similar rough texture but provides a broader view, suggesting high-quality graphene with enhanced electrical and mechanical properties. The nanoscale features indicate beneficial structural characteristics from synthesis or treatment. Figure 4(c), at 9000x magnification with a 100 nm scale bar, shows small, uniformly distributed spherical particles, indicating controlled synthesis conditions. Figure 4(d), at 10000x magnification with a 50 nm scale bar, highlights the smooth surface and uniform size of SnO_2 nanoparticles, confirming their high purity and homogeneity. The SEM image of the G/ SnO_2 nanocomposite in Figures 4(e-f), at 9000x magnification with a 20 nm scale bar, shows SnO_2 nanoparticles distributed across graphene sheets form a well-integrated structure, providing a matrix for SnO_2 nanoparticles. At 10000x magnification with a 100 nm scale bar, the image shows SnO_2 nanoparticles as uniformly distributed bright spots on the graphene surface. This uniform dispersion enhances the composite's electrical and mechanical properties, ensuring efficient charge transfer and reducing electron-hole recombination, for improving DSSCs performance.

3.5 Energy Dispersive Spectroscopy (EDS) of the synthesized samples

Figure 5(a) shows the EDX spectrum of graphene with 63.20% carbon by weight and 65.22% by atomic percentage, confirming graphene presence. Oxygen accounts for

Table 2: Summary of the electrical characteristics of G, SnO₂ NPs and G/SnO₂ NCs

Parameter	Length (cm)	Area (cm ²)	Current (μA)	Voltage (V)	Resistance (kΩ)	Sheet Resistance (Ω)	Resistivity (Ωm)	Conductivity (Sm ⁻¹)
G	1.65	6.48	3.03	0.63	207.92	641.98	8.173	0.122
SnO ₂ NPs	1.65	6.48	2.38	0.62	260.50	803.09	10.22	0.098
G/SnO ₂ NCs	1.65	6.48	3.65	0.64	175.34	549.48	6.773	0.148

20.20% by weight and 22.12% by atomic percentage, suggesting surface oxidation. Minor elements like aluminium, iron, silicon, and potassium indicate minor elements present due to method of synthesis. Figure 5 (b) shows the EDX spectrum of SnO₂ nanoparticles with 76.25% tin by weight and 75.64% by atomic percentage, confirming SnO₂ composition. Oxygen makes up 18.45% by weight and 20.24% by atomic percentage. A small amount of carbon, likely from coating of surface for SEM analysis, is also detected. The EDX spectrum of the G/SnO₂ nanocomposite (Figure 5(c)) shows significant amounts of carbon (17.25% by weight, 17.24% by atomic percentage) and tin (60.05% by weight, 60.14% by atomic percentage), confirming successful integration of SnO₂ nanoparticles into the graphene matrix. Oxygen is 3.00% by weight and 3.22% by atomic percentage. Silicon, iron and sodium, present in smaller amounts, may be residues from the synthesis.

3.6 Current versus voltage characteristics of G, SnO₂ NPs and G/SnO₂ Nanocomposite

The four-point probe (4 PP) technique measured the electrical properties of G, SnO₂ NPs, and G/SnO₂ NCs. The probe distance was 1.65 cm, and the sample's cross-sectional area was 6.48 cm². Figure 6 shows current and voltages for graphene and modified graphene thin films, with electrical parameters summarized in Table 2. Graphene electrical characterization shows a current of 3.03 μA, a voltage of 0.63 V, and a resistance

of 207.92 kΩ. The sheet resistance is 641.98 Ω, resistivity is 8.173 Ωm, and conductivity is 0.122 Sm⁻¹, indicating good electrical conductivity due to its structure and high electron mobility. The high conductivity of graphene makes it ideal for flexible electronics, sensors, and energy storage. SnO₂ nanoparticles, with a current of 2.38 μA and voltage of 0.62 V, have higher resistance (260.50 kΩ) and lower conductivity (0.098 Sm⁻¹) than graphene. The synergistic combination of graphene and SnO₂ nanoparticles yields a composite material (G/SnO₂ NCs) with superior electrical performance. By leveraging the conductivity and stability of SnO₂ and the efficient charge transfer of graphene, the composite demonstrates enhanced conductivity and lower resistivity compared to its individual components. This makes G/SnO₂ NCs an ideal material for high-efficiency photoanodes in dye-sensitized solar cells (DSSCs), where exceptional electrical performance is critical.

The nanocomposite's high conductivity (0.148 Sm⁻¹) and low sheet resistance (549.48 Ω) enhance charge transport and reduce recombination. Graphene's high electron mobility and large surface area, combined with SnO₂ semiconducting properties, create a synergistic effect for better charge separation and transport. The improved electrical characteristics, including lower resistivity (6.773 Ωm) and higher conductivity, make the graphene/SnO₂ nanocomposite promising for high-performance DSSCs, for enhancing solar cell efficiency [36].

4. Conclusion

In conclusion, this study successfully demonstrated the facile preparation and characterization of a novel graphene/SnO₂ nanocomposite (G/SnO₂ NCs) photoanode for dye-sensitized solar cell (DSSC) applications. Leveraging a sustainable approach, biomass-derived graphene was integrated with SnO₂ nanoparticles synthesized via a green route. This unique combination resulted in a significant improvement in electrical conductivity and a substantial reduction in sheet resistance compared to pristine SnO₂ and graphene individually. This performance boost can be attributed to the synergistic interplay between the high electron mobility of SnO₂ and the exceptional conductivity of graphene, which facilitates efficient charge transport within the photoanode. These findings underscore the potential of utilizing biomass-derived materials and green synthesis methodologies for developing high-performance, sustainable photoanode materials for DSSCs. Future research directions include optimizing the G/SnO₂ NCs composition to further enhance performance, rigorously evaluating its photoelectrochemical performance within fully assembled DSSC devices, and conducting comprehensive investigations into its long-term operational stability. This work lays a promising foundation for the development of next-generation, environmentally friendly solar energy conversion technologies.

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