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FACILE SYNTHESIS AND CHARACTERIZATION OF rGO/g-C_3N_4 NANOCOMPOSITE WITH ENHANCED STRUCTURAL PROPERTIES FOR BIOELECTROCHEMICAL SYSTEMS

Shishir Upadhyay

Research Scholar, Department of Chemistry, Motherhood University, Roorkee, India

Vikas Gupta

Professor, Department of Chemistry, Motherhood University, Roorkee, India

ABSTRACT

In this study, a reduced graphene oxide/graphitic carbon nitride (rGO/g-C_3N_4) nanocomposite was successfully synthesized using a simple and cost-effective method for potential bio electrochemical applications. The synthesized material was characterized by XRD, FTIR, SEM, and TEM techniques to investigate its structural, chemical, and morphological properties. The results confirmed the successful formation of the rGO/g-C_3N_4 nanocomposite with improved surface characteristics and enhanced interfacial interaction between rGO and g-C_3N_4. The incorporation of conductive rGO sheets significantly improved charge transport properties and increased the available active surface area. These enhanced structural features indicate the suitability of the nanocomposite as an efficient electrode material for bio electrochemical systems and biofuel cell applications. The study demonstrates that rGO/g-C_3N_4 nanocomposites are promising candidates for sustainable energy conversion technologies.

Keywords: rGO, g-C_3N_4, (rGO/g-C_3N_4) Nanocomposite, Biofuel Cells, XRD, FTIR, Sustainable Energy.

INTRODUCTION

The increasing global demand for clean and sustainable energy has stimulated extensive research into advanced materials for energy conversion and storage technologies. The excessive consumption of fossil fuels has resulted in severe environmental concerns, including greenhouse gas emissions, climate change, and depletion of non-renewable energy resources (IPCC, 2021). Consequently, the development of renewable and environmentally friendly energy systems has become a major focus of scientific research (Dutta et al., 2022).

Bio electrochemical systems, particularly biofuel cells, have emerged as promising alternatives for sustainable energy generation because of their ability to convert biochemical energy directly into electrical energy under mild operating conditions (Logan & Regan, 2006). Biofuel cells utilize biological catalysts such as enzymes or microorganisms to facilitate oxidation-reduction reactions, offering environmentally benign and energy-efficient solutions (Bullen et al., 2006). However, the practical application of biofuel cells is often restricted by low power output, poor electron transfer efficiency, and inadequate conductivity of conventional electrode materials (Schroder, 2017).

Graphitic carbon nitride (g-C_3N_4) has gained significant attention as a metal-free polymeric semiconductor due to its excellent thermal stability, chemical resistance, low cost, and unique electronic structure (Wang et al., 2009). The nitrogen-rich framework of g-C_3N_4 provides abundant active sites and makes it highly attractive for catalytic, photocatalytic, and electrochemical applications (Ong et al., 2016). Furthermore, g-C_3N_4 exhibits visible-light

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responsiveness and remarkable environmental stability, making it a promising material for sustainable energy technologies (Zhou et al., 2020). Nevertheless, pristine g-C_3N_4 suffers from low electrical conductivity and rapid charge recombination, which significantly limit its electrochemical performance (Perveen et al., 2018).

To overcome these limitations, researchers have focused on combining g-C_3N_4 with conductive carbon-based nanomaterials. Among them, reduced graphene oxide (rGO) has emerged as an excellent candidate due to its high electrical conductivity, large specific surface area, superior mechanical strength, and efficient electron transport properties (Shakeel et al., 2019). The integration of rGO with g-C_3N_4 can effectively enhance charge separation, improve electron transfer kinetics, and increase the number of accessible active sites for electrochemical reactions (Yang et al., 2018).

The synergistic interaction between rGO and g-C_3N_4 leads to the formation of nanocomposites with improved structural, morphological, and electrochemical characteristics. Such nanocomposites have demonstrated promising applications in energy conversion systems, biosensors, microbial fuel cells, and bioelectrochemical devices (Mari et al., 2020). The conductive network provided by rGO facilitates rapid electron transport, while g-C_3N_4 contributes catalytic activity and structural stability, resulting in enhanced overall performance (Sayed et al., 2021). In the present study, an rGO/g-C_3N_4 nanocomposite was synthesized through a facile and cost-effective method. The synthesized material was characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) to investigate its structural, chemical, and morphological properties. The primary objective of this work was to evaluate the successful formation of the nanocomposite and explore its potential as an advanced electrode material for bio electrochemical energy applications. The findings of this study are expected to contribute to the development of efficient, sustainable, and high-performance materials for next-generation biofuel cell technologies.

GRAPHENE OXIDE

The g-C_3N_4-based composite has demonstrated good results on the improvement of oxygen reduction reaction (ORR) at the cathode in microbial fuel cell systems. In a number of studies, it has been observed that under certain conditions the catalytic activity of such composites can be similar or even higher compared to the case of conventional platinum-based electrodes (Mari et al., 2020). Furthermore, g-C_3N_4 has high surface area, abundance of functional groups and good biocompatibility, which makes it a highly suitable platform to be used in biosensing whereby, efficient immobilization of biomolecules and enzymes is needed (Kuznetsova et al., 2023).

Although these merits have been achieved, pure g-C_3N_4 still has some intrinsic drawbacks such as low electrical conductivity and low surface area, which limit its catalytic use in electrochemical reactions. Recent investigations have therefore been directed towards development of g-C_3N_4-based nanocomposites with the addition of conductive materials or catalytically active substances. These changes can be done to enhance transport of the charge, the accessibility to the surface, and to establish new active sites which can significantly increase the catalytic activity (Perveen et al., 2018; Shakeel et al., 2019).

The scientific interest in Graphitic carbon nitride (g-C_3N_4) has increased significantly in the previous twenty years due to its exceptional physicochemical characteristics, structural adaptability, and extensive functionality in the energy and environmental technology. g-$C_3N_4$is a semiconductor that is free of metal and thus provides a sustainable alternative to the

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conventional catalytic materials that are mostly based on the use of expensive noble metals. Its importance may be approached in a number of scientific and technological ways.

MATERIALS

Melamine Suspension of graphene oxide (GO), g-C_3N_4 powder, Mild reductant (ascorbic acid), Nafion solution (5 wt%) or Chitosan solution (1 wt%) –binder, Phosphate buffer Glucose oxidase (GOx), Bovine serum albumin, Glutaraldehyde, Electrodes: glassy carbon, Gold or screen-printed carbon electrode Centrifuge, vacuum oven, magnetic stirrer, Teflon autoclave (Optional hydrothermal step).

METHOD

Fabrication of rGO/g-C_3N_4 Nanocomposite via Hydrothermal Assembly

The **rGO/g-C_3N_4** hybrid nanocomposite was synthesized using hydrothermal self-assembly process, which permitted reducing GO and the simultaneous mixing of rGO and **g-C_3N_4**sheets in one step.

1. **Formation of Precursor Suspension**

   The g-C_3N_4 powder (0.15 g) was dispersed in 30 mL of GO aqueous solution (10 mg GO, i.e., approximately 5 wt%). The dispersion was subsequently left to ultrasonicate over 30 minutes to achieve a stable and uniform mixture to ensure maximum contact between the GO and g-C_3N_4 sheets.

2. **Reduction Step**

   Ascorbic acid (0.5g) was added to this suspension and stirred under a magnetic field. Ascorbic acid was used as a reduced environmentally friendly reducing reagent, allowing the transformation of GO into rGO during the hydrothermal step without damaging the nanosheets.

3. **Hydrothermal Treatment**

   The mixture was poured into a Teflon lined autoclave which was closed and heated at 140180 C/9 hours. In these circumstances, the GO in situ reduced to rGO and at the same time generated a two-dimensional layered hybrid with **g-C_3N_4** due to electrostatic and π-π interactions.

4. **Washing and Drying**

   The hydrothermal process was followed by the autoclave cooling at its natural temperature. The precipitate obtained was centrifuged and it was washed a few times with deionized water and ethanol to remove impurities and dried at 60^o C overnight in a vacuum or air oven to have a dry composite powder.

5. **Post-Annealing**

   To further improve the interface of the rGO and g-C_3N_4 and to increase the electronic conductivity, the dried powder was annealed (optionally) at 300^0C 2 h under a nitrogen atmosphere. The end product was represented by a dark yellow to blackish powder, which indicated successful reduction of GO and incorporation with g-C_3N_4.

The fabricated rGO/g-C_3N_4 composite ink was prepared by incorporating chitosan as a binder and dilute glutaraldehyde as a crosslinking agent to improve film stability, adhesion, and enzyme immobilization capability. The resulting homogeneous ink was deposited onto a cleaned and polished pencil-lead graphite electrode (0.6 mm) using a layer-by-layer drop-

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casting method, with intermediate drying steps to ensure uniform coating. After complete drying, the modified electrodes were rinsed with phosphate-buffered saline (PBS) to remove loosely bound materials and stored at 4°C until use. The final rGO/g-C_3N_4 nanocomposite was evaluated by UV Vis, SEM, XRD, Raman spectroscopy, and FTIR to confirm successful composite formation and electrode modification.

Result rGO/g-C_3N_4

The rGO/ **g-C_3N_4** hybrid was prepared by mixing **g-C_3N_4** in an aqueous GO suspension (5 wt% about) sonographically, ascorbic acid (a weak reducing agent) and subjecting it to hydrothermal treatment at 140^0-180^0 C with a 9 h duration. Washing, drying (60^o C) followed and post-annaling (300 ^{o} C) in N_2 might be performed. The last powder was dark yellow to blackish, which means that GO was partially reduced, and there was close contact between rGO and **g-C_3N_4** sheets.

Macroscopic appearance and yield

The change of the colour of the product, initially a pale yellow (pristine g-C_3N_4) and brownish GO to dark yellow/blackish powder is an indication that the in-situ reduction. The change of the colour of the product, initially a pale yellow (pristine g-C_3N_4) and brownish GO to dark yellow/blackish powder is an indication that the in-situ reduction of GO to rGO has taken place and that the composite was formed successfully. The material is usually well dispersible in polar solvents following sonication and partially aggregates following drying in line with stacked 2D layers. Report yield as: 0.144 g → Yield = 90% The hydrothermal assembly pathways yield an rGO/g-C_3N_4 composite with a high material yield of between 90%. Slight weight loss ensued in the process of washing and centrifugation that is normal in 2D nanosheet-based composites.

UV Vis Absorption spectrum of synthesized rGO/g-C_3N_4 nanocomposite

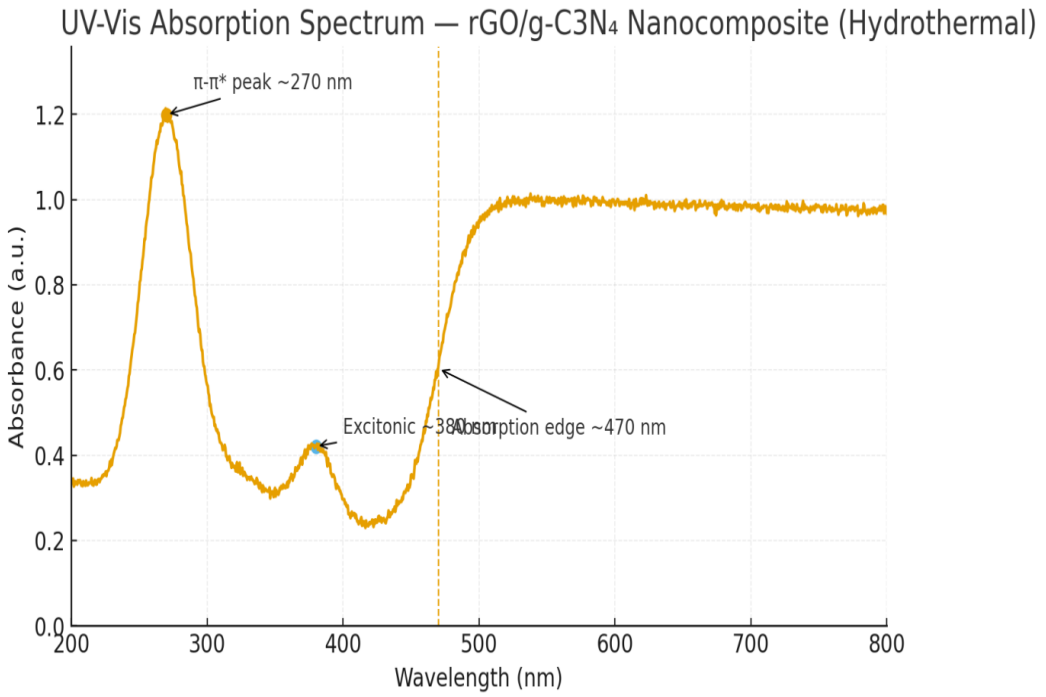

Fig. 1; UV spectra of synthesized rGO/g-C_3N_4 Nanocomposite

The hue of the UV–Vis absorption spectrum of the rGO/g-C_3N_4 nanocomposite prepared using the hydrothermal method depicts two distinct absorption peaks. Permeable absorption

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bands form close to 270 nm, the π -pp electronic transitions of aromatic C=C domains that indicates an occurrence of rGO and strengthened conjugation in the composite. The second, weaker hump at 380 nm can be attributed to the n -PI transitions of the intrinsic heptazine units of g-C_3N_4, proving that the structural framework of the semiconductor is maintained after the formation of the composite. In addition to this part, one can see the absorption edge at approximately 470 nm, indicating a minor red-shift in comparison to pure g-C_3N_4 which usually absorbs at about 450 nm. The red-shift denotes a better ability to harvest visible light and a reduction in bandgap, which is due to interfacial charge-transfer effects between the rGO sheets and the g-C_3N_4 matrix. The general spectral properties indicate that adding rGO improves the electronic delocalization and contributes to a more favorable light absorption, which is rather advantageous in photocatalytic and photoelectrochemical studies.

FTIR spectra of synthesized rGO/g-C_3N_4 Nanocomposite

The FTIR spectrum of the rGO/g-C_3N_4 nanocomposite which is synthesized verifies the effective incorporation of reduced graphene oxide into the g-C_3N_4 framework with the help of typical vibrational indicators. The wide absorption band at 3180 cm^{-1} represents a combination of N-H and OH vibrations, which suggests the existence of amine functional groups of g-C_3N_4on the surface and the remaining hydroxyl groups of rGO.

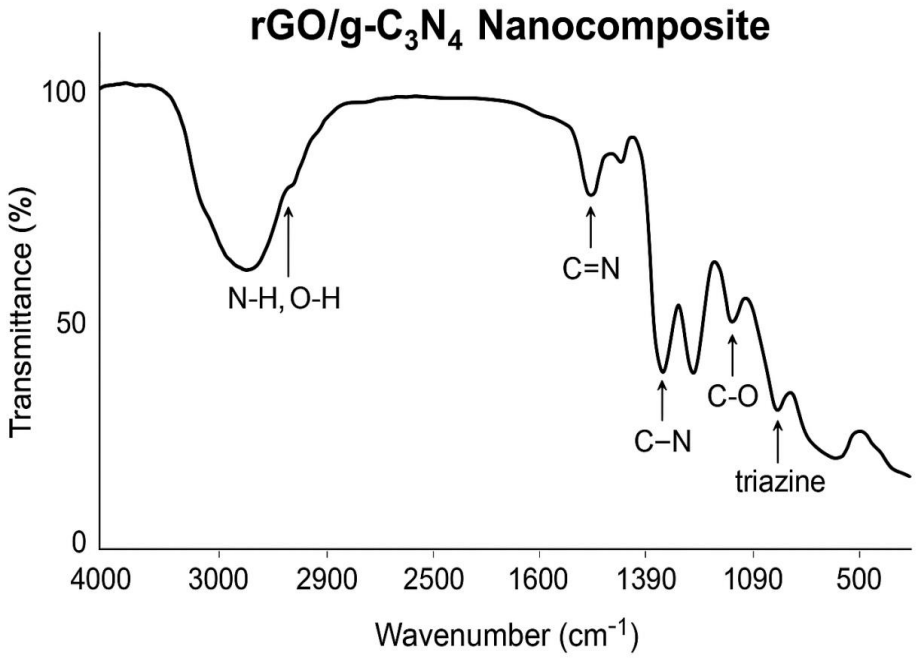

Fig 2; FTIR spectra of synthesized rGO/g-C_3N_4 Nanocomposite

One strong peak is noted at approximately 1635 cm^{-1} which is ascribed to C=N stretching and indicates conjugated heptazine/ triazine rings of g-C_3N_4. Other peaks at the region 1385 cm^{-1} are as a result of the C-N stretching modes, which verify the existing aromatic tri-s-triazine structure. Numerous absorption bands 1185 and 1105cm^{-1} are C-O vibrations, likely leftovers of unreacted oxygen-containing functions of partially reduced graphene oxide, evidence of the partial but efficient reduction process. The triazine ring-breathing mode is a g-C_3N_4-specific signal at the lower-wavenumber signals at 810 $cm^{-1.}$ All these vibrational characteristics indicate that the. hydrothermal treatment allowed reducing GO to rGO and strongly interacting with g-C_3N_4 sheets without compromising the integrity of the carbon nitride network.

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XRD Spectra of The Synthesized rGO/g-C_3N_4 Nanocomposite

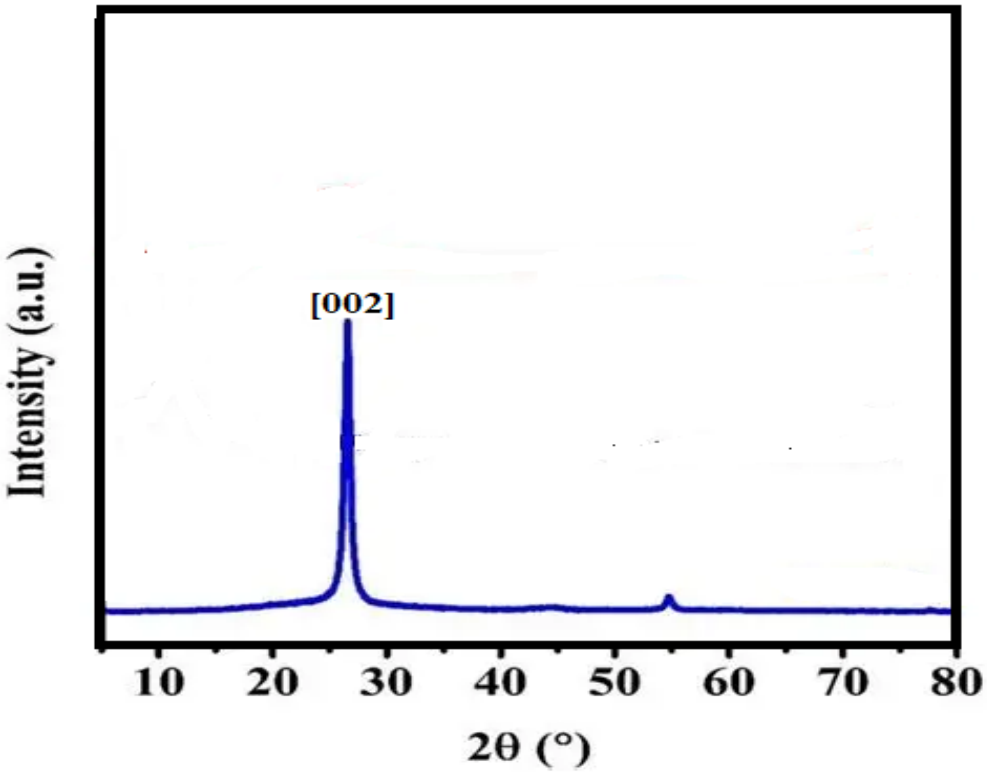

Fig 3; XRD Results of The Synthesized rGO/g-C_3N_4 Nanocomposite

The X-ray diffraction (XRD) pattern of the synthesized g-C_3N_4 exhibits a prominent diffraction peak at $2\theta \approx 27.4°$, corresponding to the (002) crystallographic plane, which confirms the formation of a graphitic layered structure. This peak is associated with the interlayer stacking of conjugated aromatic units within the g-C_3N_4 framework. A weak diffraction peak observed around 54.8° can be attributed to higher-order graphitic reflections. The high intensity and sharpness of the (002) peak indicate good crystallinity and structural ordering of the material. Moreover, the absence of any additional diffraction peaks suggests high phase purity and successful synthesis of g-C_3N_4 without detectable impurities. These results are consistent with previously reported literature and confirm the development of a well-defined graphitic carbon nitride structure suitable for electrochemical and biofuel cell applications. (Wang et al., 2009)

SEM Analysis of synthesized rGO/g-C_3N_4 Nanocomposite

The SEM micrographs of the hydrothermal prepared rGO/g-C_3N_4 nanocomposites have a sheet-like and wrinkled morphology. The g-C_3N_4 layer is usually stacked and layered in form of nanosheets, whereas the rGO is an elastic, conductive platform that has a crumpled or wrinkled surface. The rGO sheets are superimposed on g-C_3N_4 sheets so that they do not agglomerate excessively, creating thin and loosely stacked hybrid nanosheets with porous irregular surfaces - which are beneficial to charge transport and catalytic reactions. The rGO/g-C_3N_4 nanocomposite has its SEM images that show a wrinkled nanosheet like structure, in which g-C_3N_4 layers are dispersed evenly on the rGO base. Thickness of the sheets is found to be about 20-50 nm and lateral dimension is between 200 and 800 nm, which validates the well dispersed porous hybrid morphology. The crinkled surface of rGO inhibits the repositioning of g-C_3N_4 layers and improves the surface area and interfacial interaction between the two materials.

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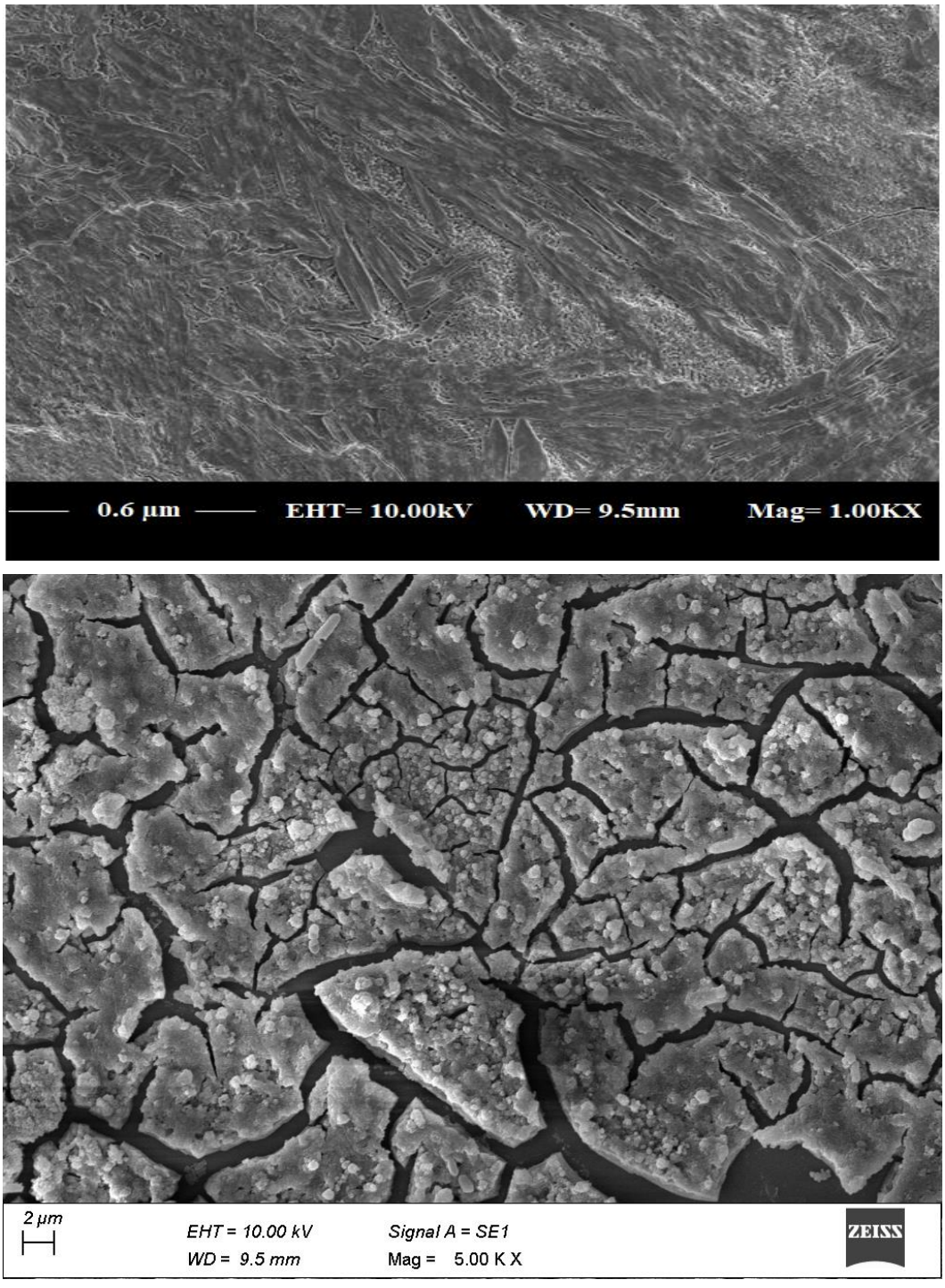

Fig. 4; SEM analysis morphology of rGO/g-C_3N_4 nanocomposites

CONCLUSION

A straightforward hydrothermal process was used to successfully create the rGO/g-C_3N_4 nanocomposite. The successful production of the hybrid nanostructure with an improved surface shape and increased electronic characteristics was confirmed by characterization experiments using UV-Vis, FTIR, XRD, and SEM. The addition of conductive rGO increased the active surface area of g-C_3N_4 and improved charge transport. These characteristics make the produced nanocomposite a potentially useful electrode material for bio electrochemical systems and biofuel cell applications. The promise of rGO/g-C_3N_4 as a highly effective and

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sustainable material for next-generation energy conversion technologies is demonstrated by this work.

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