

Studies on Optical Band Gap and Optical Conductivity of *Ex-situ* Fabricated rGO:V₂O₅ Composites

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Abstract: In this present study, Graphene oxide (GO) is synthesized by modified Hummer's method. The GO formed is further used to prepare V₂O₅ embellished reduced graphene oxide (rGO) composites (rGO: V₂O₅) in varying ratios via an in-situ process. UV-Vis spectroscopy was employed to study optical properties such as band gap and optical conductivity of the composites and individual species. rGO synthesized was studied to have a narrow band gap of 1.93 eV, which was much lower than the metal oxide used. Thus, rGO enticing material is to be used to modify the band gap of the prepared composites, thus lowering the optical band gap to 1.32 eV for rGO: V₂O₅ (2:1) and enhancing the optical conductivity to 5.34×10¹⁰ S.cm⁻¹. Characterization of the compound is done using various analytical techniques, including FTIR, SEM, XRD, and TGA. All these studies designate the successful fabrication of the required composites with specific properties. Thus, it is concluded from the study that rGO:V₂O₅ in 2:1 can be used as a potent material in optoelectronic applications.

Keywords: Clean and affordable energy, optical conductivity, optical band gap, reduced graphene oxide, V₂O₅.

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1. Introduction

In recent years, the study of the optical properties of material has been an interesting area of research due to the increasing demand for advanced materials with unique electronic and optical properties [1]. In current studies, rGO and V₂O₅ synthesize a hybrid material with better optical activity than the individual material.

V₂O₅ was chosen for the research due to its remarkable properties, such as good adsorption in visible regions, electrical conductivity, and great electrochemical performance. Along with this, V₂O₅ is an easily available and efficacious material [2-4]. rGO has been used due to its ability to act as a supporting material for charge transport. rGO consists of many important properties, such as electrical conductivity, large surface area, and good optical properties [5-8].

Various studies have been done to investigate the optical properties of rGO: V₂O₅ composites that are obtained by the *ex-situ* fabrication method. One of the important optical properties of these materials is the optical band gap, which measures the energy required to excite the electrons from the valance band to the conduction band. This is a significant property

for designing and developing devices like solar cells, photodetectors, and light-emitting diodes [9-10].

Optical conductivity is a property of the material that determines its ability to conduct light. Knowing the optical conductivity of rGO : V₂O₅ composites can optimize their optical performance for numerous applications. Thus, it is an important area to research as it can potentially contribute to developing advanced optical and electronic devices [11-13]. In this research work, rGO is composited with V₂O₅ in different ratios to study the synergetic impact of V₂O₅ and rGO with each other. The exciting results make these composites a promising contender for optoelectronic applications.

2. Materials and Methods

2.1. Materials.

Graphite, Conc. Sulphuric acid (H₂SO₄), Potassium permanganate (KMnO₄), Ascorbic acid, Hydrochloric acid (HCl), 30% Hydrogen peroxide (H₂O₂), and V₂O₅ are purchased from Loba Chemie Pvt. Ltd.

2.2. Synthesis of GO from graphite.

GO is synthesized from graphite by using Modified Hummer's method. 5g of Graphite powder is taken in a 500 ml beaker, to which 75 ml of concentrated H₂SO₄ is added. 15 g of KMnO₄ is added very slowly to the reaction. The material was stirred for 2 hours at a maintained temperature of 5°C. Afterward, the temperature increased to 35°C, and the material was kept vigorously stirred for 30 minutes. 200 ml deionized water is added to the above solution, which generates a temperature rise from 35-98°C owing to the heat of hydration. Stirring is continued for the next 45 minutes at the temperature mentioned above. 140 ml deionized water and 20 ml H₂O₂ (30 wt. %) is then added, which results in the generation of yellow-brown precipitates of GO. Further, on vacuum filtration, the precipitates are separated and washed with 5% aqueous HCl. The obtained filtrate was dried for 24 hours at 60°C in a vacuum oven [14-15].

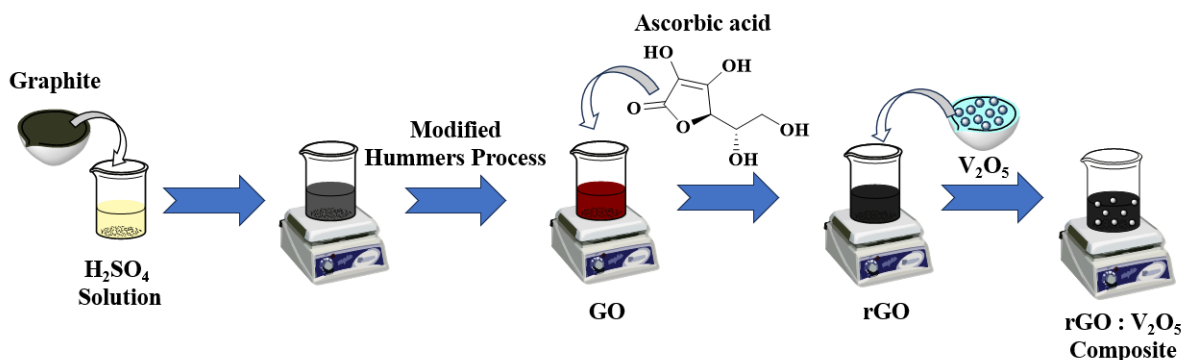
2.3. Synthesis of rGO by ascorbic acid.

GO is transformed to rGO with the help of ascorbic acid. 1g of GO prepared earlier is dispersed in 400 ml of water. 10 g of ascorbic acid is added to the solution and is allowed to stir for 120 minutes at 60°C. The product is obtained as a thick black slurry and is collected with the help of centrifugation. 10-20 ml H₂O₂ (30 wt. %) was added to the reaction mixture, and it was stirred for 30 minutes at 60°C to remove excess ascorbic acid. A black-colored product is obtained and washed with ethanol and water 3-4 times. The product is collected with the help of centrifugation and dried at 120°C for 24 hours [16-17].

2.4. Preparation of composite ratios.

Composite of rGO and metal oxide V₂O₅ are prepared in varying ratios of 1:1, 1:2, and 2:1 using the solvothermal method. rGO is prepared by reducing GO with the help of ascorbic acid. V₂O₅ is added to the rGO formed, and stirring is continued for 1 hour at 60°C (Scheme 1). The product is sonicated for 30 minutes at room temperature for better insertion of V₂O₅ in

the rGO layers. The product is obtained by centrifugation followed by decantation. The ratios are dried at 60°C and ground to powder form [18].



Scheme 1. Diagrammatic representation of the preparation of rGO : V₂O₅ composite.

3. Results and Discussion

3.1. UV-Vis spectroscopy.

UV-Vis spectroscopy is used to study the optical properties of the compounds. Characterization of compounds is done by using ethanol as a solvent. The instrument used for the analysis is a Shimadzu UV-1900i spectrometer with a wavelength range of 200-800 nm. According to the data collected with the help of UV-Vis spectroscopy, the peaks observed at 245 nm in GO are slightly shifted to 279.78 nm in rGO. This peak shift shows the rearrangement of graphene sheets after the reduction process [19]. The peaks observed in GO at 245 nm and 310 nm are of π to π^* and n to π^* transitions due to the presence of C=C bonds and C=O bonds, respectively. As GO is reduced to rGO, a red shift is observed [20-21] (Figure 1).

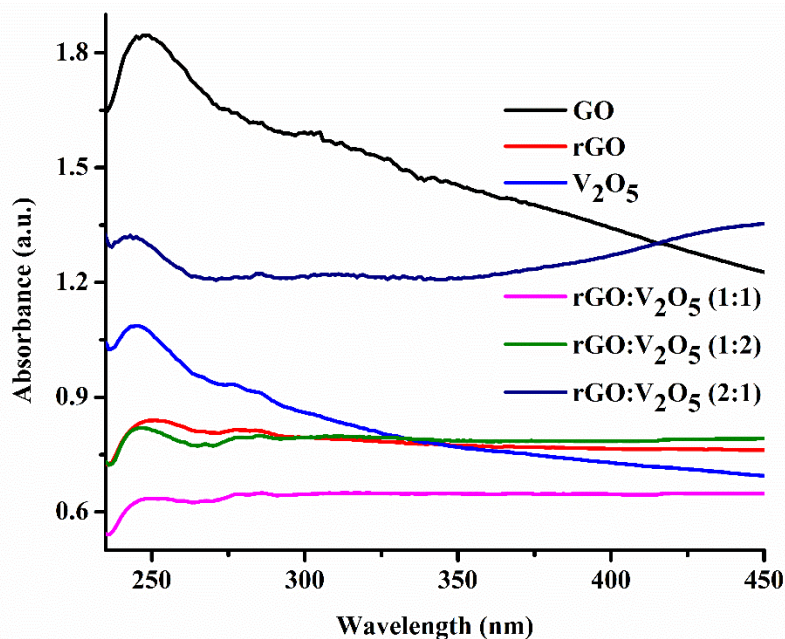


Figure 1. UV-Vis absorption spectra of all individual and composite materials.

Band gap is an intrinsic property of an optically conductive material; the band gap is observed for different ratios of the rGO:V₂O₅ composites, V₂O₅, and rGO with the help of UV-Vis spectroscopy. For rGO, the observed band gap lies near 1.93 eV, while for V₂O₅, the band

gap lies around 2.82eV (Figure 2a); the results follow the reported studies [22-23]. When the band gap of composites was studied, a decrease in the band gap of metal oxide V₂O₅ was seen for 1:1 and 2:1 samples. It is expected as such due to the introduction of defects in the composites leading to an increase in charge carrier density and mobility. Whereas in 1:2 sample, an increase in the band gap is observed. It may be due to the higher band gap of transition metal oxide V₂O₅.

Optical conductivity is calculated for all the samples. It has been observed that optical conductivity for V₂O₅ and rGO is 2.07×10¹⁰ and 3.55×10¹⁰ S.cm⁻¹, respectively (Figure 2 b). The optical conductivity for the rGO:V₂O₅ composites that were studied has been observed in various ratios of composites (Table 1). The formulas used for calculating optical conductivity are as follows:

Transmittance (T):

$$T = \exp(-2.303 A) \dots \dots \dots (1)$$

Reflectance (R):

$$T = (1 - R)^2 \exp(-A) \dots \dots \dots (2)$$

Reflectance has been used to calculate the reflective index (n) with the help of the following formula:

$$n = \left(\frac{4R}{(R-1)^2} - K^2 \right)^{1/2} - \frac{(R+1)}{(R-1)} \dots \dots \dots (3)$$

Extinction coefficient (K):

$$k = \frac{\lambda \alpha}{4\pi} \dots \dots \dots (4)$$

Optical conductivity(σ):

$$\sigma = \frac{anc}{4\pi} \dots \dots \dots (5) [24]$$

Table 1. Observed band gap and optical conductivity of rGO, V₂O₅, and synthesized composites.

Compound	Observed band gap (eV)	Optical conductivity×10 ¹⁰ (S.cm ⁻¹)
rGO	1.97	3.55
V ₂ O ₅	2.82	2.07
rGO : V ₂ O ₅ (1:1)	2.34	2.16
rGO : V ₂ O ₅ (1:2)	3.21	1.30
rGO : V ₂ O ₅ (2:1)	1.32	5.34

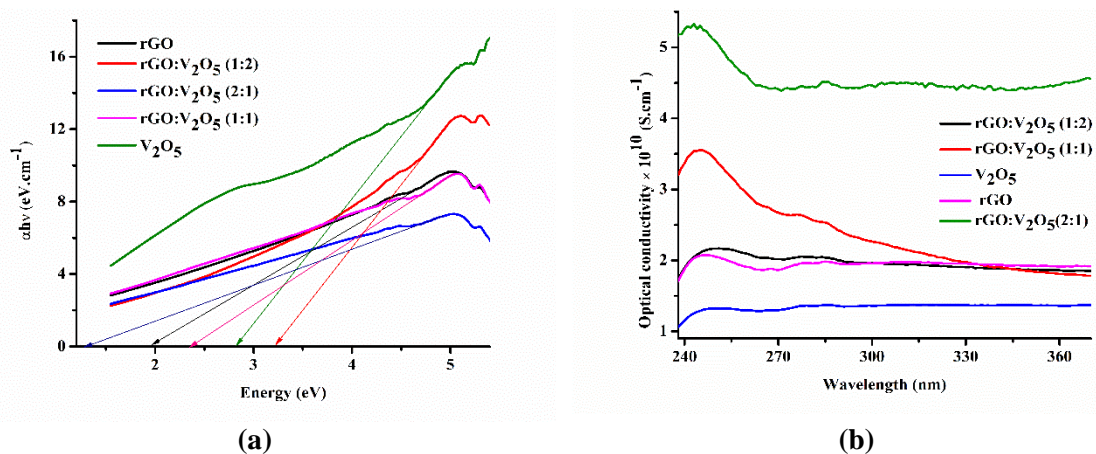


Figure 2. (a) Band gap of rGO, V₂O₅, and synthesized composites; (b) Optical conductivity of rGO, V₂O₅, and synthesized composites.

3.2. Fourier transforms infrared (FTIR) spectroscopy.

The Fourier transform infrared spectra were observed with the help of KBr pellets using a Perkin Elmer spectrometer with a Diamond ATR detector ranging from around 400 to 4000 cm^{-1} with a resolution of 1 cm^{-1} .

3.2.1. FTIR of GO.

The FTIR spectra of GO (Figure 3) show the peaks 1063 cm^{-1} , which refers to the C-O stretching. The peak observed at 1224.85 cm^{-1} confirms the C-O-C bending. Peak near 3342.37 cm^{-1} refers to OH stretching vibrations of C-OH group and water content. Peak observed near 1700 cm^{-1} shows the C=O stretch, and peaks near 1623 cm^{-1} show the C=C stretch [25].

3.2.2. FTIR of rGO.

In the IR spectra of rGO (as shown in Figure 3), the peaks observed in the GO, 1224, 3342, 1623, and 1700 cm^{-1} are either removed or decreased, displaying that GO has been reduced to rGO. A peak near 1066 cm^{-1} shows the presence of a C-O stretch in the compound. The peak near 1600 shows the presence of a C=C bond [26].

3.2.3. FTIR of V_2O_5 .

In the IR spectra of V_2O_5 (as shown in Figure 3), the peak is observed near 1003, 830.23, 513.35, and 464 cm^{-1} . Peaks observed near 1003 cm^{-1} represent stretching vibrations for terminal bonds of oxygen (V=O). Peaks near 513.35 cm^{-1} and 464 cm^{-1} represent symmetric and asymmetric stretches of triply coordinated oxygens. Peaks observed near 830.23 cm^{-1} represent vibrations of bridged oxygen [27].

3.2.4. FTIR of rGO: V_2O_5 .

The peaks observed in Figure 3, near 3318 cm^{-1} and 1699 cm^{-1} , represent the O-H stretching and CO stretching, respectively. The peak observed near 1560 cm^{-1} represents C=C stretching. The peaks observed near 1003 cm^{-1} show stretching vibrations for terminal bonds of oxygen (V=O). Since rGO: V_2O_5 composite has peaks similar to both rGO and V_2O_5 , It contributes to the characterization of the formation of the required composite [26-27].

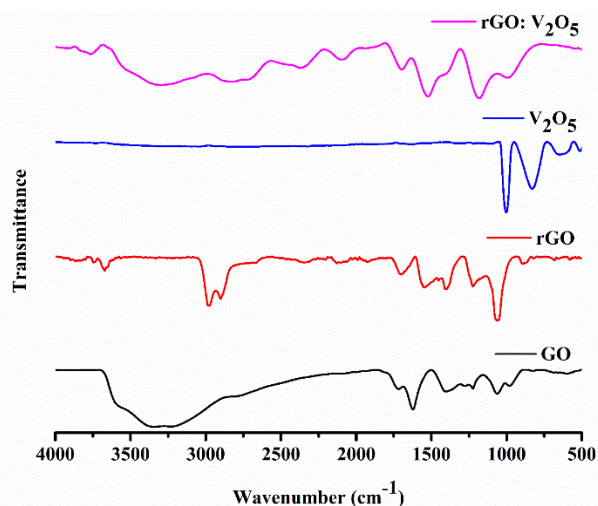


Figure 3. FTIR spectra of all the materials.

3.3. X-ray diffraction analysis.

X-ray diffraction (XRD) is a great analytical technique that helps predict a compound's material properties. The XRD machine used to characterize the samples is Bruker AXS D8 Advance A25-X1-1A2Z2C4B0. The spectra are recorded at a 2θ range of $10^\circ - 70^\circ$, with the help of an anode copper source of Cu-K α radiation ($\lambda=1.54 \text{ \AA}$) at the temperature of 25°C . In Fig. 4, a sharp peak was observed at $2\theta=10.5^\circ$, and a weak peak near 21° suggests the formation of GO. The peak near $2\theta = 10^\circ$ is disappeared in rGO due to the reduction process. The peak observed near 23° and 43° shows the reduction of GO to rGO. Since it is a broad peak, it is signified that crystallization has not occurred properly, and the compounds are amorphous. In Fig. 4, the peaks observed at 15.9° , 20.2° , 22.03° , and 31.1° refer to the orthorhombic phase of V_2O_5 . Since the peaks observed are sharp, it suggests the presence of the crystalline nature of V_2O_5 [28]. In Fig. 4, the peaks were observed near 23° and 43° . Due to the interaction between V_2O_5 and rGO, the peaks present in rGO are intensified [29].

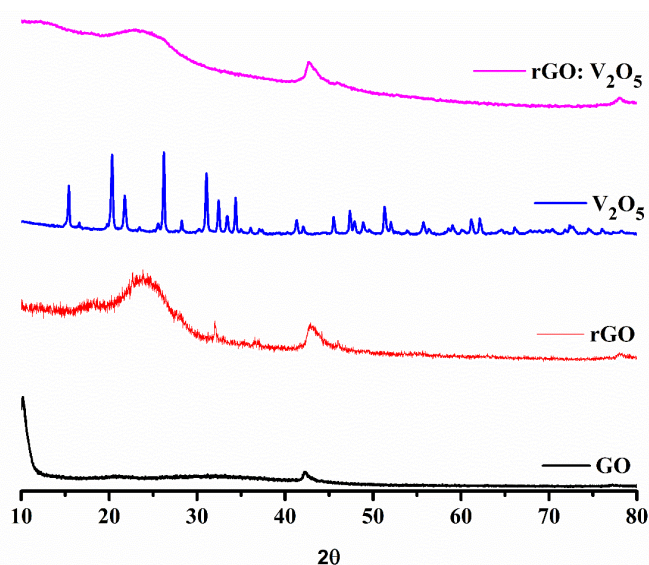
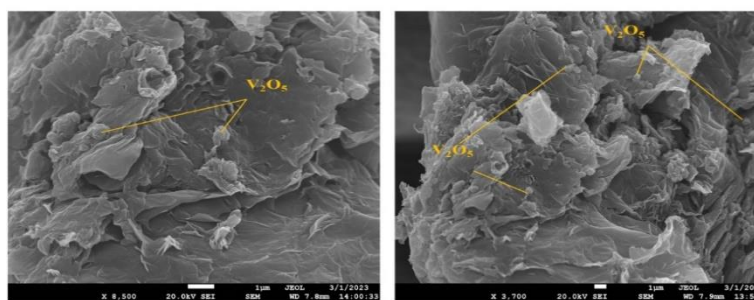


Figure 4. X-ray diffraction pattern for all the materials.

3.4. Scanning electron microscopy (SEM).

Scanning electron microscopy (SEM) is a technique used to study the morphology of compounds. The study of composite morphology is done using FE-SEM instrument model FESEM: JSM-7610F-Plus, Au Sputter Coater: DII-29030SCRT. Samples are coated with gold before the analysis. The scanning electron microscopy image of rGO: V_2O_5 composite is represented in Figure 5. The flake-like structures present in the FE-SEM images represent rGO. V_2O_5 particles present on the surface of rGO are observed to be agglomerated with each other, forming a bead-like structure [30].



a)

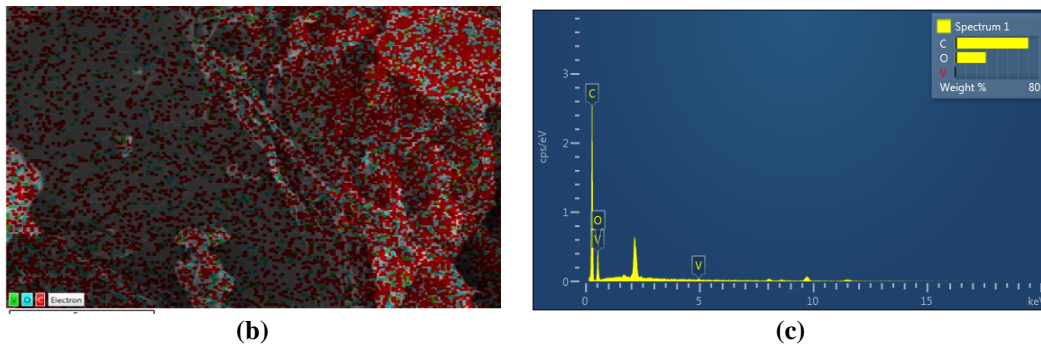


Figure 5. (a) Images of scanning electron microscopy of rGO: V₂O₅ (2:1) indicating the presence of V₂O₅ in the matrix of rGO; (b) EDS Layered image of FESEM rGO: V₂O₅ (2:1) representing the presence of expected elements; (c) Energy dispersive X-ray spectra of rGO: V₂O₅ (2:1).

3.5. Thermogravimetric analysis (TGA).

Thermogravimetric analysis (TGA) is an analysis technique used to measure the thermal stability of a compound. For the current studies, the instrument used is the Perkin Elmer thermal analyzer. The temperature range is between 75-580°C in nitrogen flow at a heating rate of 10°C per minute. Figure. 6 represents the TGA curve of V₂O₅, rGO, and rGO: V₂O₅ (2:1) composite. In rGO, 16.25% weight loss was observed till 200°C. 200-400°C contributes to a weight loss of 37.09%, which is further lost to 44.58% till 515°C. V₂O₅ reduces 18% of its weight to 200°C, 37.5% to 400°C, and 46% to 515°C. In the case of composite rGO: V₂O₅, weight loss is 0.2% to 200°C, 0.7% to 400°C, and 1.05% to 515°C [27, 31].

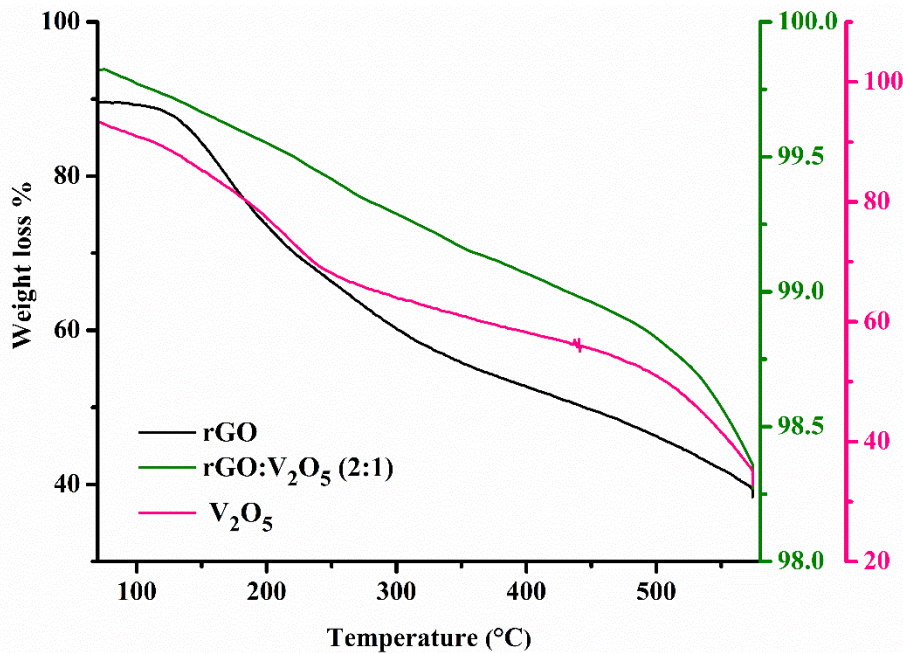


Figure 6. Thermogravimetric analysis of rGO, rGO:V₂O₅, V₂O₅.

4. Conclusions

Based on the current studies on optical band gap and optical conductivity of *ex-situ* fabricated rGO: V₂O₅, a great reduction in band gap is observed. This reduction is attributed to the introducing of defects in the rGO: V₂O₅ composites. rGO itself is a good material because it has less band gap. When it is combined with V₂O₅, an increase in carrier concentration and mobility is observed. Due to the reduction in band gap (1.32 eV) and increase in optical conductivity (5.34×10^{10} S.cm⁻¹) for rGO: V₂O₅ (2:1), the composites can be used for applications like solar cells and photodetectors.

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Conflicts of Interest

The authors declare no financial or personal conflict of interest influences the work reported in this paper.

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