



Synthesis and characterization of rGO/CoSnO₃ and rGO/NdSn₂O₇ nanocomposites for Photocatalytic application

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ABSTRACT

Reduced graphene oxide wrapped cobalt tin oxide and neodymium tin oxide nanocomposites synthesized via facile hydrothermal method. The synthesized rGO/CoSnO₃ and rGO/NdSn₂O₇ nanocomposites were characterized by various analytical techniques such as XRD, FT-IR, SEM, XPS and UV-DRS. The phase and crystalline size of GO, rGO, Co₃O₄, CoSnO₃, NdSn₂O₇, rGO/CoSnO₃ and rGO/NdSn₂O₇ are found to be 19.8, 27.6, 26.9, 37.3, 42.8, 40.5 and 46.2 nm respectively. The elemental composition and ionic state was examined by using XPS analysis. Surface texture and morphology were examined by using SEM which revealed that CoSnO₃ and NdSn₂O₇ agglomerated nanoparticles are uniformly distributed over the surface of rGO with a 2D sheet structure. The band gap was calculated using UV-DRS analysis and the Kubelka - Munk function plot scrutinized that band gap of rGO/CoSnO₃ and rGO/NdSn₂O₇ was found to be 2.7 and 2.5 eV. Herein, we have employed methylene blue dye for the photocatalytic degradation purpose using rGO/CoSnO₃ and rGO/NdSn₂O₇ nanocomposite and achieved up to 92% degradation property. This research outcome might be useful for reducing water pollution.

Introduction

Environmental pollution has been a major problem encountered in recent years. The level of pollutants is increased day by day, causing severe and irreparable damage to the globe from the surface of the earth[1]. Industrial effluents have been identified as the major source of water contamination. Photocatalytic degradation is an important waste water treatment process that can eliminate harmful heavy organic contaminants [2]. Secondary effects of traditional wastewater treatment methods include the high cost of separation and the generation of secondary pollutants, associated with adsorption, clotting and membrane separation involving high operational costs[3]. Graphene has been regarded as a promising support material for energy storage batteries which can improve charge transfer and mass transport. Graphene oxide is also enhanced by photocatalytic degradation property[4]. CoSnO₃ and NdSn₂O₇ is a transition metal oxide semiconductor with wide-direct band gap. CoSnO₃ and NdSn₂O₇ nanostructures have attracted great attention for potential applications and also various fields such as photocatalytic performance, gas sensor, glucose sensor and solar cells. Generally metal oxides have a better photocatalytic efficiency [5,6]. In

particular rGO/NdSn₂O₇ acts as the best photocatalyst with high stability. As a result, the evolution of the new and efficient nanocomposite is progressively essential due to the environmentally beneficial product. In this study rGO/CoSnO₃ and rGO/NdSn₂O₇ and were used to estimate the degradation of methylene blue dye under sunlight irradiation and the observed results were studied and analyzed.

Experimental Materials

In this study the required chemicals such as Graphite powder, Sodium nitrate, Sulphuric acid, Potassium permanganate, Tin chloride (SnCl₂.2H₂O), Cobalt chloride (CoCl₂.2H₂O), Neodymium nitrate Nd(NO₃)₃.6H₂O, Oxalic acid and Hydrogen peroxide were in the analytical grade and used as such without any further purification.

Synthesis of Graphene Oxide

Graphene oxide was synthesized by modified Hummer's method. 46 mL of H₂SO₄ and 1 g of graphite powder and sodium nitrate were taken in the beaker and the solution was stirring for one hour for 500 rpm[7]. 6 g of potassium permanganate was added slowly and the solution was stringing half an hour in



ice bath condition at 37 °C for 30 minutes. Further, 50 mL of water was added drop wise, and cooling the prearrangement in room temperature, then the arrangement was warming on 100 °C and 10 mL of hydrogen peroxide was added for the formation of graphene oxide[8].

Synthesis of Reduced Graphene Oxide

400 mg of powdered graphene oxide (0.1 mg/mL) was dissolved in 20 mL of water and hydrogen hydrate (10 mL) was added and it used as a reducing agent to reduce the graphene oxide. The mixture was centrifuged at 4000 r/s for 40 minutes after being put on a magnetic stirrer for 30 minutes at 60 °C. Then, the mixture was washed with ethanol and distilled water and dried for 24 hours at 120 °C[9].

Synthesis of Cobalt Oxide

In a typical synthesis, cobalt oxide is synthesized via hydrothermal method. 1.6 g of cobalt chloride and 0.6 g of oxalic acid were dissolved in 70 mL of water. It was stirred continuously for half an hour at room temperature. The mixture was transferred into Teflon lined stainless steel autoclave and maintained at 120 °C for 12 h. The product was washed with water and ethanol to remove unwanted impurities and dehydrated at 80 °C [10]. The resultant material was annealed at 400 °C for 5 h in a muffle furnace.

Synthesis of CoSnO₃ and rGO/CoSnO₃ Nanocomposite

1.6g of Cobalt chloride and 0.6 g of sodium hydroxide were dissolved in water. 0.9 g of tin chloride was added slowly. The solution was stirred continuously for half an hour at room temperature. The obtained mixture was transferred into Teflon lined stainless steel autoclave and maintained at 120 °C for 12 hrs[11]. The product was washed with water and ethanol, removed to remaining ions, and dehydrated at 80 °C in air. The resulting powder was annealed at 5 hrs for 450 °C in a muffle furnace to get CoSnO₃. The above-mentioned pathway was used to synthesize rGO/CoSnO₃ nanocomposite. The powdered rGO is added with the obtained CoSnO₃ to get rGO/CoSnO₃ nanocomposite[12].

Synthesis of NdSn₂O₇ and rGO/ NdSn₂O₇ Nanocomposite

To synthesis rGO/NdSn₂O₇ via one-step hydrothermal method, 2.2 g of tin chloride and 0.3 g of sodium hydroxide were added into 50 ml of distilled water and 4.38 g of Neodymium nitrate was added into the solution to maintain the pH value at 7. The mixture of the solutions kept in magnetic stirring for 30 minutes at room temperature[13]. The mixed solutions were transferred into a 100 ml Teflon lined stainless steel autoclave heated at 160°C for 10 hours. After that the sample was washed with ethanol and water and the product was dried at 80 °C in hot air oven. The resulting powder was annealed at 5 hrs for 450 °C in a muffle furnace to get NdSn₂O₇. The above-mentioned pathway was used to synthesize rGO/NdSn₂O₇ nanocomposite. The powdered rGO added with the obtained NdNiO₃ to get rGO/NdSn₂O₇ nanocomposite[14].

Results and Discussion XRD Analysis

The crystalline size and phase of the materials were characterized via., XRD analysis. The XRD patterns of GO and rGO observed diffraction peaks at 2θ values of 16, 26 and 45° found in the planes of (002) and (001) respectively(Fig.1a) [15]. Fig.1b shows the intense diffraction peaks of Co₃O₄ nanoparticle it is around the 2θ values of 36.23, 59.17 and 64.28° correspond to the (220), (422) and (511), planes respectively. These 2θ values are well matched with the JPCDS card no. 73-1702. 2θ values of CoSnO₃ composite are observed at 33.14°, 39.05, 46.21, 58.72 and 66.30° and the values are ascribed to the reflection plane of (211), (220), (400), (422) and (511), it is well matched with JCPDS Card No. 29-0514[16]. The 2θ values Nd₂Sn₂O₇ are observed at 27.13, 32.24, 35.67, 42.32 and 46.82° and the values ascribed the planes of (100), (222), (001),(400) and (440) and the values are well matched with JCPDS Card No. 13-0185. These phenomena indicate that the formation of CoSnO₃ and Nd₂Sn₂O₇ nanoparticle began at the calcination temperature of about 450 °C[17]. Peaks are not detected in other phases, indicating the high purity of the products shown in Fig.1. The different parameters are calculated, such as the crystallite size of rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposites using Debye–Scherrer formula (Equation-1) and the values are presented in Table 1.

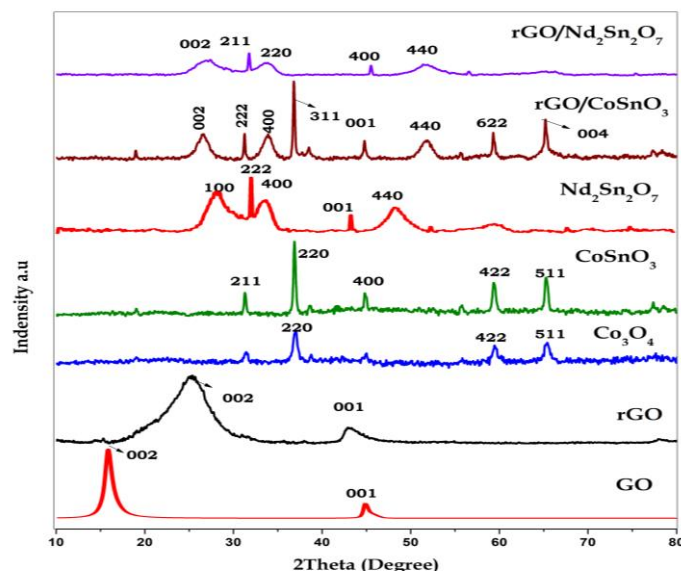


Fig. 1. XRD pattern of GO, rGO, Co₃O₄, CoSnO₃, Nd₂Sn₂O₇, rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposites

Debye–Scherer’s equation

$$\text{Crystalline size (D)} = \frac{0.9\lambda}{\beta \cos \theta}$$

----- (1)

Where λ is the wavelength ($\lambda = 1.5406 \text{ \AA}$ (Cu K α)), β is the full width half maximum (FWHM) and θ is the diffraction angle.

Table.1. Crystalline size of rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposite

S. No	Sample	Crystalline Size(nm)
1.	GO	19.8
2.	rGO	27.6
3.	Co ₃ O ₄	26.9
4.	CoSnO ₃	37.3
5.	Nd ₂ Sn ₂ O ₇	42.8
6.	rGO/CoSnO ₃	40.5
7.	rGO/Nd ₂ Sn ₂ O ₇	46.2

FT-IR Spectrum of rGO/CoSnO₃ Nanocomposite

The FT-IR technique is a strong tool to analyze the different functional groups found in graphene oxide, including functional groups involving oxygen. FT-IR spectra of rGO (**Fig. 2a**) shown the absorption stretching vibrations peaks, centered at 1594 cm^{-1} , which can belong to the stretching of C=O groups, while the vibration band observed at 3458 cm^{-1} is due to the stretching mode of H-O-H group of rGO. The stretching vibrations peaks of 566 cm^{-1} and 665 cm^{-1} is due to the stretching mode of the Co-O bond (**Fig. 2b**) [18]. The pure CoSnO₃ and the characteristic bands at large attributed to the H-O-H band is appeared at 3419

cm^{-1} show in **Fig. 2c** and good agreement with the reported values [19]. In the rGO/CoSnO₃ composite band at 540 cm^{-1} due the metal-oxygen stretching vibration and this co-existence may be significant(**Fig. 2d**). The **Fig. 2e** indicate the FT-IR spectrum of Nd₂Sn₂O₇, the metal-oxygen band appeared at 665 and 688.5 cm^{-1} and the XRD pattern of rGO/Nd₂Sn₂O₇ is shown in **Fig. 2f**, H-O-H band appeared at 3480 cm^{-1} and the composite metal-oxygen stretching vibration band appeared at 596 and 688 cm^{-1} since it prevents the combination of charge carriers and also induces a synergistic effect to enhance catalytic activity of the nanocomposite[20].

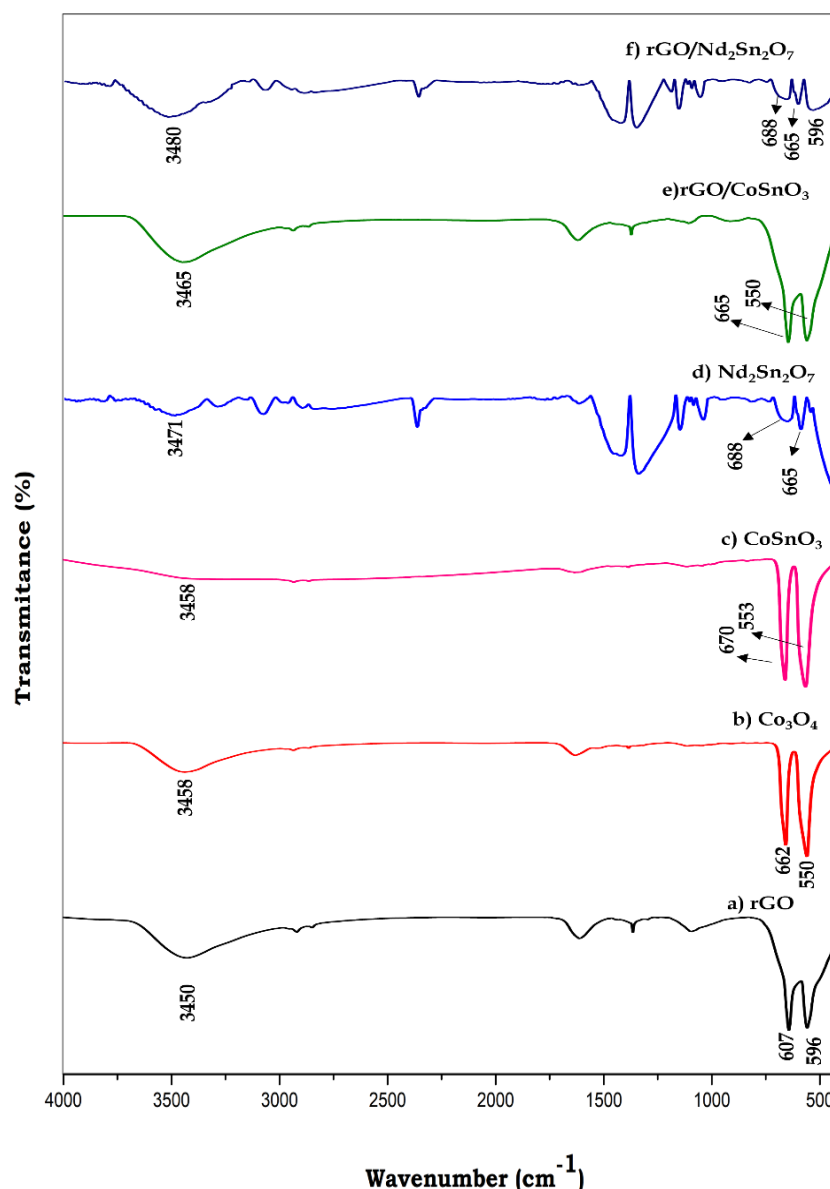


Fig. 2. FT-IR spectra of rGO, Co₃O₄, CoSnO₃, Nd₂Sn₂O₇, rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposites

XPS Analysis of rGO/CoSnO₃ nanocomposite

Ionic state and chemical composition of the prepared materials were characterized through XPS analysis. The XPS spectra of the prepared rGO/CoSnO₃ nanocomposite were shown in **Fig. 3**. As from the **Fig. 3a**, the survey spectrum of rGO/CoSnO₃ nanocomposite was illustrated the presence of Co, Sn, O and C elements in the survey spectrum [21]. Moreover, no other impurities were found in the spectrum rather than rGO/CoSnO₃ nanocomposite-based elements. **Fig. 3a- Fig. 3e** shows the core level spectra of the Co, Sn, O

and C elements. **Fig. 3b** shows the high resolution deconvoluted spectrum of Co2p, it exhibits the two major peaks at 781.78 and 797.34 eV binding energies are corresponding to Co2p_{3/2} and Co2p_{1/2} respectively. Also, the energy separation between the two energy levels is about 16.03 eV. **Fig. 3c** shows the deconvoluted spectrum of Sn3d was exhibited at 486.7 and 495.03 eV, which are related to the Sn3d_{3/2} and Sn3d_{5/2} states and the energy spin separation was about 8.06 eV [22]. The C1s peak (**Fig. 3d**) can be deconvoluted into 3 peaks, each indicating the different



carbon bonds such as C=C (283.5 eV), C-O (284.6 eV) and C=O (286.7 eV). Further, the peak intensities of oxygen (C=O) associated groups in the C1s peak are

rather low, suggesting that a good degree of reduction has taken place where most of the oxygen functional groups have been removed.

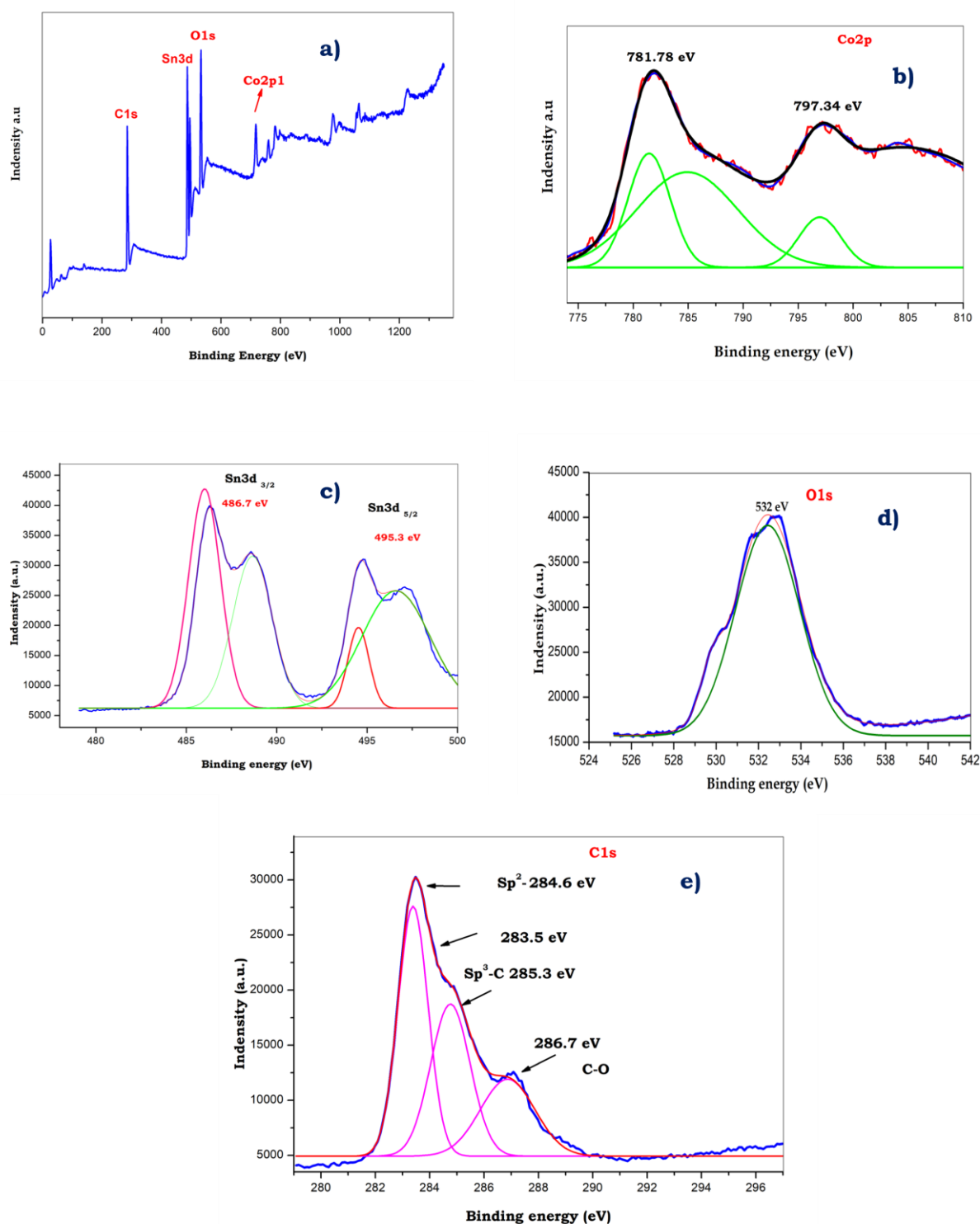


Fig. 3 XPS spectra of rGO/CoSnO₃ nanocomposite (a) Survey spectrum (b) Co2p core spectrum, (c) Sn3d core spectrum, (d) O1s core spectrum and (e) C1s core spectrum



XPS Analysis of rGO/Nd₂Sn₂O₇ nanocomposite

The detailed study of high resolution core level spectra of the Nd, Sn, C and O elements are shown in the **Fig. 4**. In **Fig. 4a**, the rGO/Nd₂Sn₂O₇ nanocomposite was exhibit Nd, Sn, C and O elements without appearing any other impurities indicating the purity of the rGO/Nd₂Sn₂O₇ nanocomposite. **Fig. 4b** shows the high resolution deconvoluted spectrum of Sn3d, it exhibits the two major peaks at 486.11 and 494.64 eV binding energies are corresponding to Sn3d_{5/2} and Sn3d_{3/2} respectively[23]. **Fig. 4c** shows the deconvoluted

spectrum of Nd3d was exhibited at 980.13 eV and 1005.27 eV corresponds to the Nd3d_{5/2} and Nd3d_{3/2} sates. The deconvoluted spectrum of the C1s state was shown in the **Fig. 4d** and it is observed at 265.13 eV. Moreover, the O1s state with the deconvoluted spectra of the rGO/Nd₂Sn₂O₇ nanocomposite was shown in the **Fig. 4e**. This peak related to the oxygen containing functional groups was present in the reduced graphene oxide. The peak at 531.02 eV corresponds to surface bonded water (H–O–H) [24].

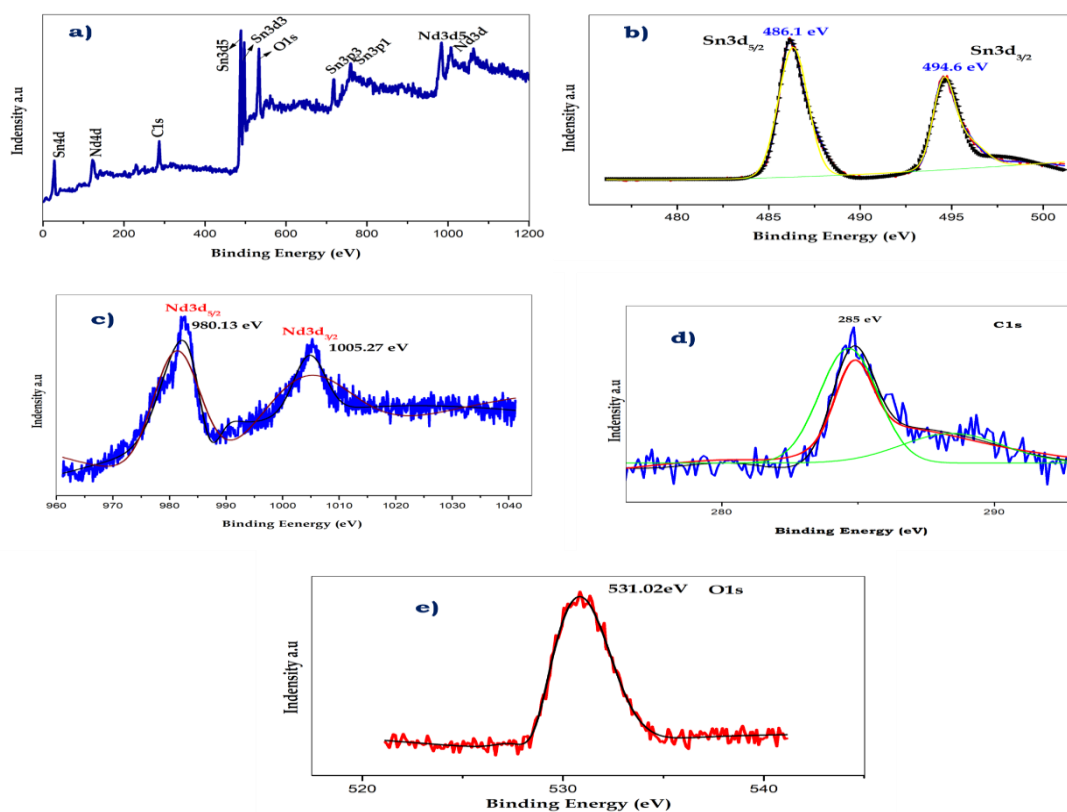


Fig. 4 XPS spectra of rGO/CoSnO₃ nanocomposite, (a) Survey spectrum (b) Sn3d core spectrum, (c) Nd3d core spectrum, (d) C1s core spectrum and (e) O1s core spectrum

Morphological analysis

The surface morphologies of the prepared nanomaterials were investigated using SEM analysis. **Fig. 5** shows the SEM images of rGO/CoSnO₃ and. **Fig. 5a and 5b** shows the SEM images of GO and rGO exhibited a sheet-like structure [25]. **Fig.5c** shows the cubic shaped morphology of Co₃O₄ nanoparticles and

Fig.5d shows the agglomerated CoSnO₃ nanoparticles[26]. **Fig.5e** denoted the agglomerated CoSnO₃ nanoparticles are distributed in rGO surface. Granular shaped Nd₂Sn₂O₇ nanoparticles showed in **Fig. 5f**. The Nd₂Sn₂O₇ nanoparticles are agglomerated on the surface of rGO (**Fig.5g**).

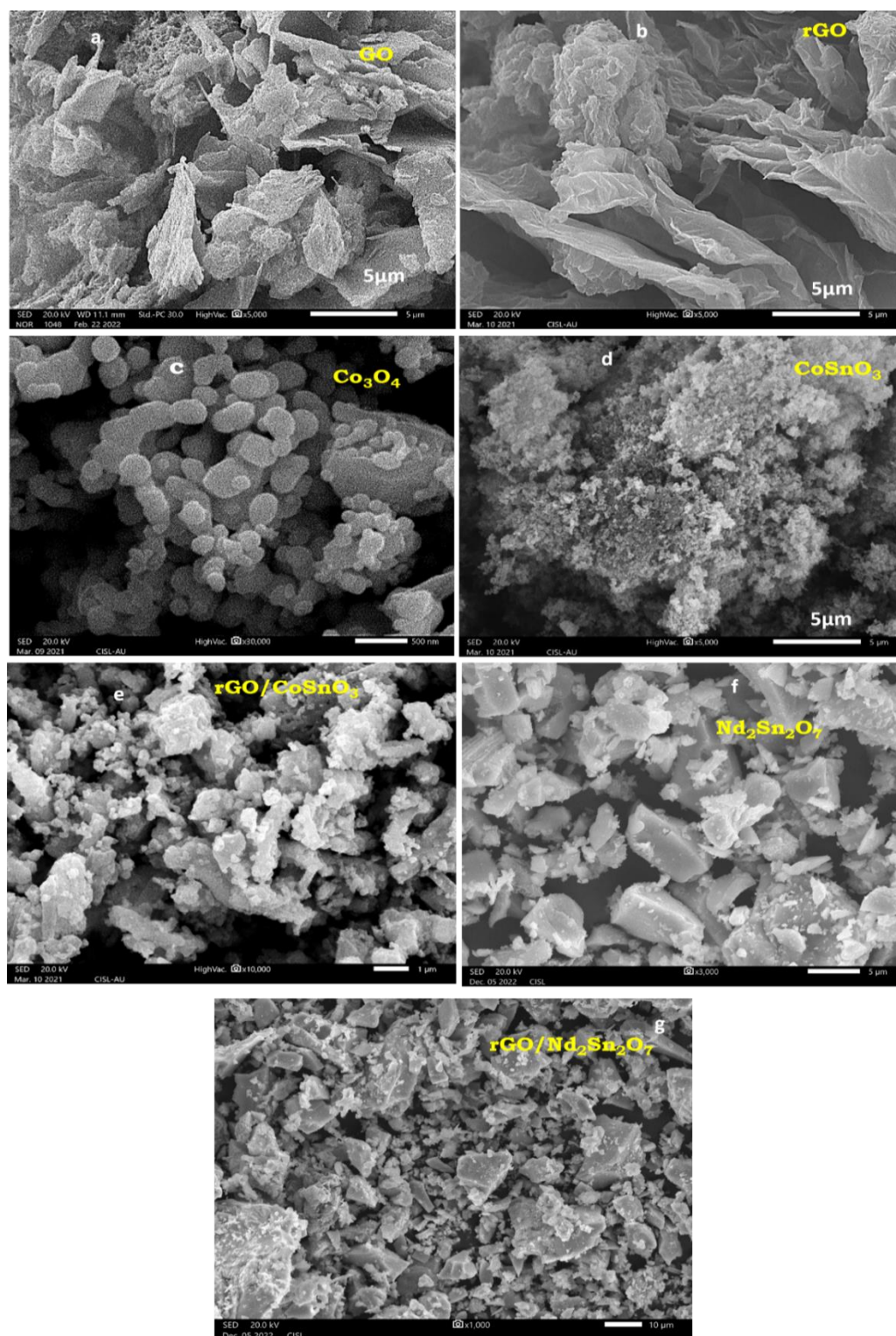


Fig. 5. a) SEM images of GO, (b) rGO and (c) Co_3O_4 , (d) CoSnO_3 , (e) $\text{rGO}/\text{CoSnO}_3$ and (f) $\text{Nd}_2\text{Sn}_2\text{O}_7$, (g) $\text{rGO}/\text{Nd}_2\text{Sn}_2\text{O}_7$ nanocomposites

(f)



UV - DRS Analysis

UV – diffuse reflectance spectroscopy of rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposites are shown in **Fig. 6** and it is illustrated that pure rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposites exhibited significant UV absorption edge observed at 400 to 800 nm. But the UV

absorption of other samples shifted towards higher wavelength side. The changes in the absorption edges show the changes in the band structure. Further, the bandgap of samples are determined by Kubelka – Munk function equation[27].

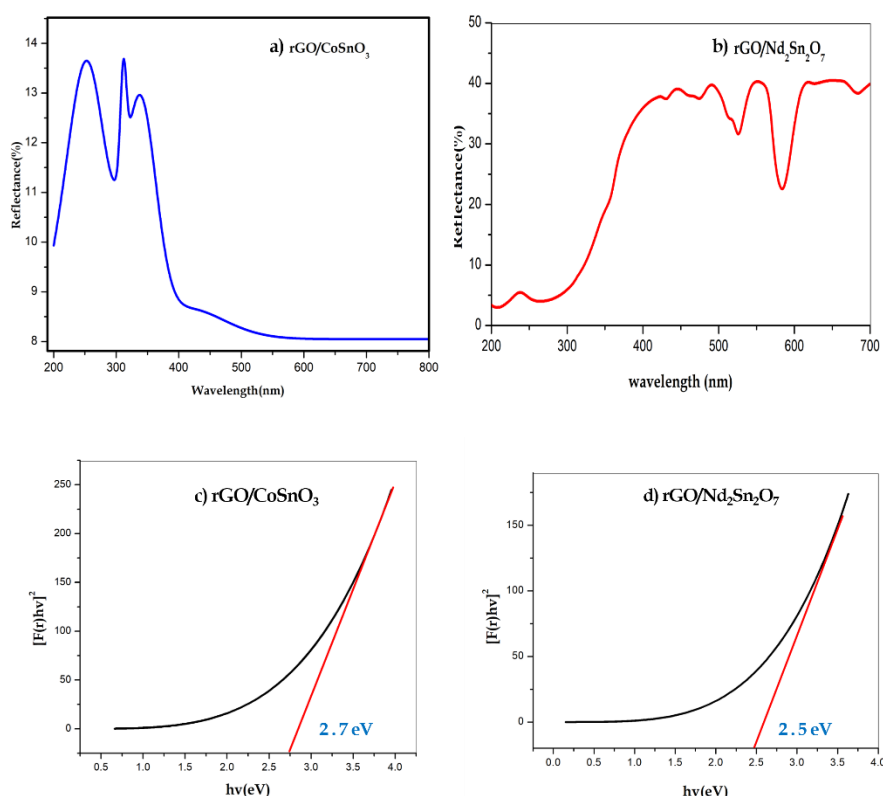


Fig. 6 UV-DRS and UV-DRS band gap images of (a) UV-DRS image of rGO/CoSnO₃, (b) UV-DRS image of rGO/Nd₂Sn₂O₇, (c) UV-DRS band gap of rGO/CoSnO₃ and (e) UV-DRS band gap of rGO/Nd₂Sn₂O₇

$$\alpha h\nu = A (h\nu - E_g)^n \text{ ----- (2)}$$

Where α is the absorption coefficient and $h\nu$ is the incident photon energy. As shown in **Fig. 6c and 6d**, the bandgap energies are estimated from the intercept of the tangents. The band gap of prepared rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposite were found to be 2.7 and 2.5 eV respectively. The presence of oxygen vacancies can create impurity level near the valance bond, the rGO/Nd₂Sn₂O₇ having lower band gap value and it is having more catalytic activity compared to the rGO/CoSnO₃ nanocomposite.

Photocatalytic degradation processes

To find the degradation kinetics of methylene blue dye, 20mg of the catalyst was added to the dye solution and the system was kept in sunlight irradiation which was stirred magnetically. The system was observed manually and the degradation time was fixed as 60 minutes for methylene blue. When the catalyst was added into the dye solutions, the absorption peaks of the dye solution started to vanish and 5 ml of the samples were collected every 10 minutes for the UV analysis. The absorption curves (**Fig.7a and Fig.7b**), degradation efficiency (%) and absorbance plots (**Fig.8**) and kinetic analysis of photodegradation and degradation efficiency plots (**Fig.9**) which explained



clearly about the degradation capability of the nanocatalysts[28]. The kinetic study for rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposite (photocatalyst) for the degradation of Methylene blue were studied by using Langmuir – Hinshelwood kinetic model by using

equation-3. The calculated rate constant of rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposite for the photodegradation of methylene blue is 0.721×10^{-4} and $1.830 \times 10^{-4} \text{ min}^{-1}$ respectively.

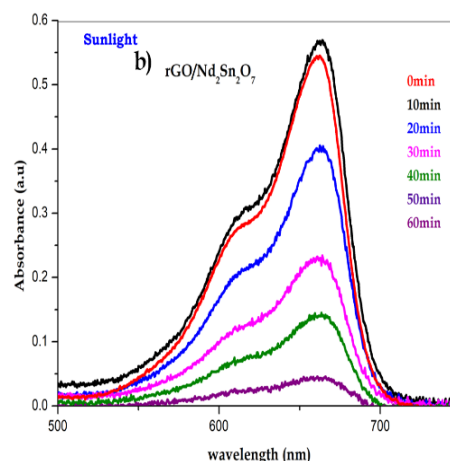
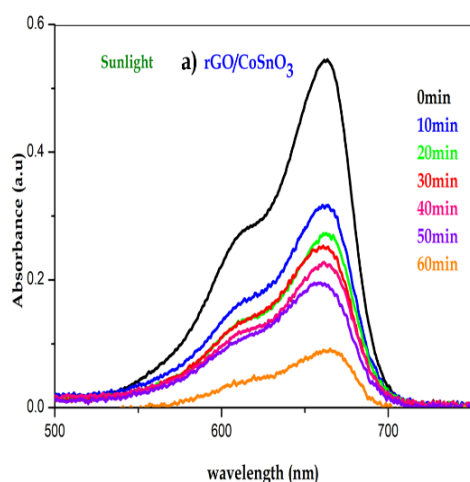


Fig. 7 Photocatalytic degradation of methylene blue dye using 20 mg of catalyst a) rGO/CoSnO₃ and (b) rGO/Nd₂Sn₂O₇.

Degradation Efficiency of rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ under Sunlight Irradiation

The degradation efficiency of the nanocomposite were carried out in weight percentage of 20 mg of the catalyst. The removal efficiency was find out with

sunlight irradiation.

Fig. 8 explain that the degradation efficiency of the prepared materials by using the sunlight irradiations and the rGO/Nd₂Sn₂O₇ material has more efficiency than the rGO/CoSnO₃ material[29].

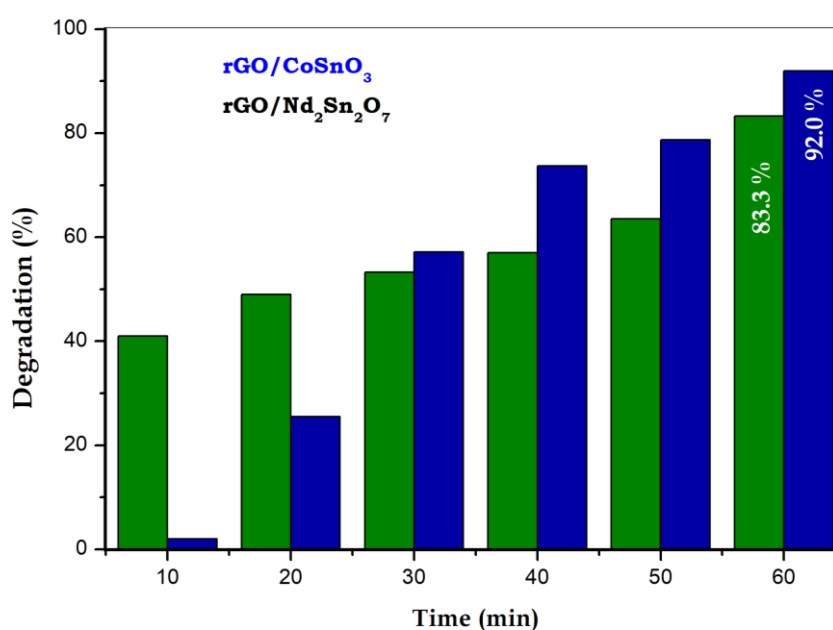


Fig. 8 Photocatalytic degradation efficiency of methylene blue dye using 20 mg of catalysts rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇.



Photocatalytic reaction kinetics

The reaction rate of photodegradation of methylene blue (MB) dye with the nanocatalysts was measured at various time intervals with 20 mg of catalyst. A linear correlation between $\ln(C_0/C_t)$ and reaction time t , was observed (**Fig.9**), indicating that the photocatalytic degradation process of methylene blue (MB) dye by rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ follows a pseudo-first order kinetic model (Eq-3).

$$\ln(C_0/C_t) = -kt \quad \dots\dots\dots (3)$$

Where “C₀” is the initial concentration (mmol/L), “C_t” is the dye instantaneous concentration at various illumination intervals, and k is the apparent rate constant. rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ are shown in **Fig.9** and the observed rate constant indicated that the rGO/Nd₂Sn₂O₇ having higher rate constant in sunlight irradiation. In the **Fig.9** denote the rate constant values of prepared nanomaterials in sunlight irradiation, 20 mg of rGO/Nd₂Sn₂O₇ having higher rate constant value (1.83 min⁻¹) in sunlight irradiation.

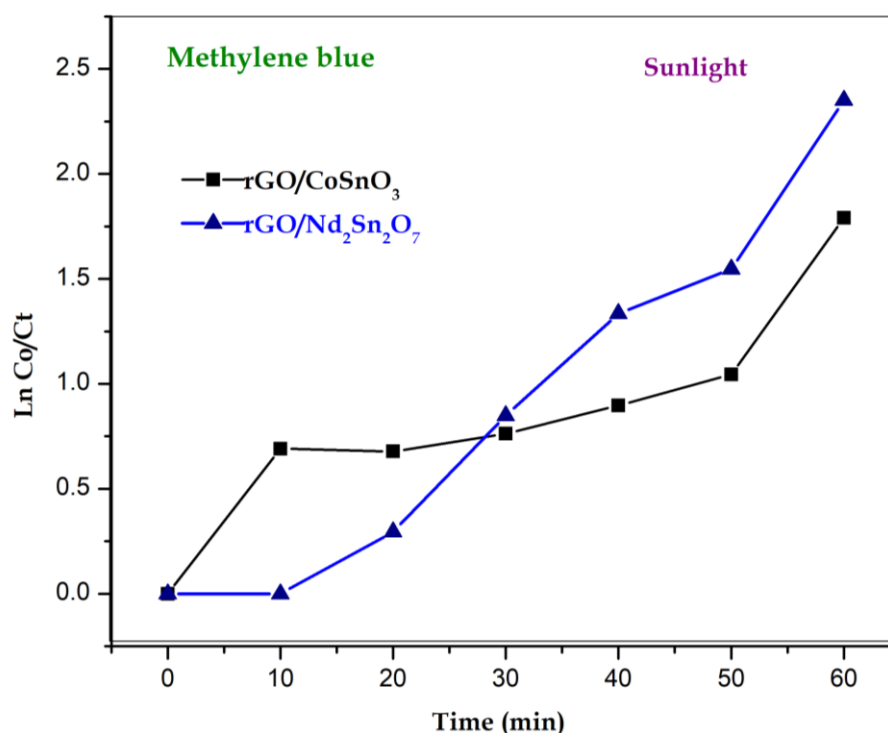


Fig. 9 Pseudo-first order plot of the rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposite under sunlight irradiation

Mechanism of photocatalysis:

When rGO/CoSnO₃ and rGO/Nd₂Sn₂O₇ nanocomposites is irradiated with light energy larger than or equal to its band gap energy, conduction band electrons (e⁻) and valence band holes (h⁺) are produced, according to the actual process of methylene blue dye degradation. The formation of hydroxyl radicals mediates the oxidation of organic compounds, whereas the production of superoxide radicals mediates the reduction and oxidation reactions[30]. **Fig.10** illustrates a schematic representation of the degradation mechanism. The idea that hydroxyl radical forms are to blame for the oxidation route of chemical compounds begun by

heterogeneous photocatalysis. The dye might be reduced or the photogenerated electrons could combine with electron acceptors like O₂ adsorbed on the rGO surface or dissolved in water, reducing it to superoxide radical anion O₂^{-•}. The photogenerated holes can oxidize organic compounds by reacting with OH⁻ or H₂O to produce ·OH radicals [31]. Because the OH· radical is such a powerful oxidizing agent, it can oxidize most MB into non-toxic end products such as CO₂, H₂O and Mineralized product.

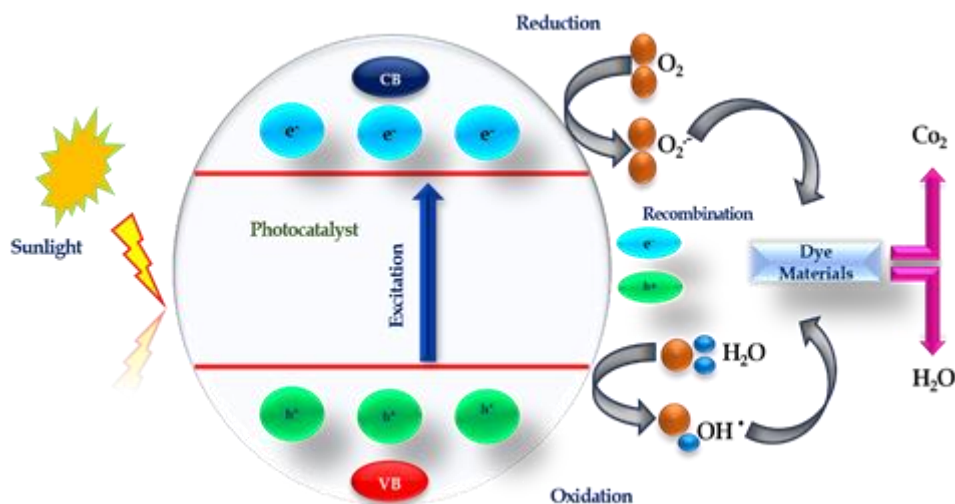


Fig.10. Mechanism of photodegradation of Methylene Blue Dye

Conclusion:

The hydrothermal approach was employed to synthesize the nanomaterials viz., GO, rGO, Co_3O_4 , CoSnO_3 and rGO/CoSnO_3 and $\text{rGO/NdSn}_2\text{O}_7$. The prepared nanocomposites were calcined at 450°C for 6 hours, producing GO, rGO, Co_3O_4 , CoSnO_3 , NdSn_2O_7 , rGO/CoSnO_3 and $\text{rGO/NdSn}_2\text{O}_7$ nanomaterials with a crystalline size of 19.8, 27.6, 26.9, 37.3, 42.8, 40.5 and 46.2 nm with the cubic and agglomerated structures. Kubelka - Munk function plot scrutinized that the band gap of rGO/CoSnO_3 and $\text{rGO/NdSn}_2\text{O}_7$ was found to be 2.7 and 2.5 eV respectively. The photocatalytic performance of the synthesized rGO/CoSnO_3 and $\text{rGO/NdSn}_2\text{O}_7$ against methylene blue dye was evaluated by sunlight irradiation. $\text{rGO/NdSn}_2\text{O}_7$ nanocomposite having a high degradation property (92%) compared to rGO/CoSnO_3 (83.3%) material. Sunlight irradiation is a preferable source for eco-friendly photocatalytic degradation processes. The materials used to reduce the water pollution via an efficient photodegradation processes.

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