

Advanced Graphene Oxide-Nickel Oxide Nano Composites: A Promising Platform for Photocatalytic Environmental Cleanup

Sandesh V. Gaikwad^{1*}, Ujjan B. Kadam¹, Bhaskar K. Nikam², Pratibha G. Raundal², Sanket D. Khairnar²

¹Department of Chemistry, Mahatma Gandhi Vidyamandir's Maharaja Sayajirao Gaikwad Arts, Commerce, and Science College, Malegaon, Maharashtra, India, ²Department of Chemistry, M.V.P. Samaj's Karmaveer Abasaheb Alias N. M. Sonawane Arts, Commerce, and Science College, Nashik, Maharashtra, India

ABSTRACT

The continuous discharge of dye-contaminated wastewater from industrial operations is a major environmental hazard, demanding the development of effective and long-term cleanup techniques. In this work, graphene oxide (GO) and GO-nickel oxide (NiO) nanocomposites were produced and comprehensively tested for photocatalytic efficiency in the breakdown of organic dyes using visible light irradiation. The structural and morphological properties of the synthesized materials were investigated using X-ray diffraction, scanning electron microscopy, and energy-dispersive X-ray spectroscopy (EDS), confirming the successful formation of GO-NiO nanocomposites with uniform dispersion of NiO nanoparticles on GO sheets. Optical investigation demonstrated that the GO-NiO nanocomposite has a lower band gap, leading to increased visible light absorption. Photocatalytic investigations revealed that the GO-NiO nanocomposite exhibited considerably greater degradation efficiency than virgin GO, owing to enhanced charge separation and reduced electron-hole recombination arising from strong interfacial contacts. The degradation process exhibited pseudo-first-order kinetics, indicating positive reaction dynamics. Furthermore, the nanocomposite demonstrated remarkable stability and reusability after many catalytic cycles. Overall, the findings demonstrate the efficacy and sustainability of GO-NiO nanocomposites as photocatalysts for environmental remediation by degrading organic contaminants in aqueous environments.

Key words: Band gap engineering, Charge separation, Electron-hole recombination, Nickel oxide nanoparticles, Photocatalytic kinetics.

1. INTRODUCTION

The rapid expansion of industrialization and urbanization has significantly increased the discharge of hazardous pollutants into the environment, particularly in the form of dye-containing wastewater [1]. Industries such as textiles, leather processing, pharmaceuticals, and paper manufacturing are major contributors to this problem [2]. Synthetic dyes are widely used due to their stability, brightness, and resistance to degradation; however, these same properties make them persistent in natural water systems [3]. Many dyes are toxic, carcinogenic, and non-biodegradable, posing severe threats to aquatic ecosystems and human health [4]. Even at low concentrations, they can reduce light penetration in water bodies, thereby affecting photosynthetic activity and disrupting ecological balance [5]. Consequently, the development of efficient and environmentally friendly methods for the removal of dye pollutants have become a critical research priority. Traditional wastewater treatment techniques, including adsorption, coagulation-flocculation, membrane filtration, and biological degradation, have been extensively employed to remove dyes from aqueous solutions [6]. Although these methods can reduce pollutant levels to some extent, they often suffer from several limitations, such as incomplete degradation, high operational cost, generation of secondary pollutants, and low efficiency for recalcitrant compounds [7]. In contrast, advanced oxidation processes, particularly photocatalysis, have emerged as highly effective alternatives for the complete mineralization of organic pollutants. Photocatalysis involves

the use of semiconductor materials that, upon light irradiation, generate electron-hole pairs capable of initiating redox reactions [8]. These reactions produce highly reactive species such as hydroxyl radicals ($\bullet\text{OH}$) and superoxide ions ($\text{O}_2^{\bullet-}$), which can degrade complex organic molecules into harmless end products such as carbon dioxide and water [9]. Among various photocatalytic materials, graphene oxide (GO) has attracted considerable interest due to its unique structural and physicochemical properties. GO is a two-dimensional carbon-based material derived from graphite, consisting of a layered structure decorated with oxygen-containing functional groups such as hydroxyl, epoxy, and carboxyl groups. These functional groups enhance hydrophilicity and provide abundant active sites for the adsorption of pollutant molecules. In addition, GO exhibits a high specific surface area, excellent mechanical strength, and good electronic conductivity,

*Corresponding Author

Sandesh V. Gaikwad,
E-mail: sandesh.628@rediffmail.com

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making it a promising candidate for catalytic and environmental applications [10]. However, the photocatalytic activity of pure GO is relatively limited due to the rapid recombination of photogenerated electron-hole pairs and insufficient visible light absorption. To overcome these limitations, the design of hybrid nanocomposites by coupling GO with semiconductor materials has gained significant attention. Transition metal oxides are widely used in photocatalysis owing to their suitable band structures, chemical stability, and cost-effectiveness. Nickel oxide (NiO), a p-type semiconductor with a wide band gap and good thermal stability, has been explored for various catalytic applications. However, NiO also suffers from limitations such as charge carrier recombination and low surface area.

The integration of GO with NiO to form GO-NiO nanocomposites provides a promising solution to these challenges. GO acts as an electron acceptor and conductive support, facilitating electron transfer and reducing recombination. This enhances the generation of reactive oxygen species and improves photocatalytic efficiency. Furthermore, the large surface area of GO increases pollutant adsorption, while NiO provides active catalytic sites. The formation of heterojunctions between GO and NiO further improves charge separation and extends light absorption into the visible region. Recent studies have reported enhanced photocatalytic activity of GO-based nanocomposites compared to individual components due to improved surface area, light harvesting, and charge carrier dynamics. However, systematic comparative studies between pure GO and GO-NiO nanocomposites under identical conditions remain limited. Therefore, the present study focuses on the synthesis and comparative evaluation of GO and GO-NiO nanocomposites for photocatalytic dye degradation under visible light irradiation. The relationship between material properties and photocatalytic performance is investigated, along with catalyst stability and reusability, to develop efficient and sustainable nanomaterials for environmental remediation.

2. MATERIALS AND METHODS

2.1. Materials

Graphite powder ($\geq 99\%$ purity) was used as the precursor for the synthesis of GO. Nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) was employed as the nickel source for the preparation of NiO. Analytical grade sulfuric acid (H_2SO_4), potassium permanganate (KMnO_4), hydrogen peroxide (H_2O_2), and hydrochloric acid (HCl) were used for the oxidation process. Sodium hydroxide (NaOH) was utilized for pH adjustment during synthesis. All chemicals were of analytical grade and used without further purification. Deionized water was used throughout the experimental work.

2.2. Synthesis of GO

GO was synthesized using a modified Hummers' method. Initially, graphite powder was added to concentrated sulfuric acid under continuous stirring in an ice bath to maintain a low temperature. Potassium permanganate was slowly introduced into the mixture while keeping the temperature below 20°C to prevent overheating. The reaction mixture was then stirred at an elevated temperature until it formed a thick paste. Subsequently, deionized water was added gradually, followed by the addition of hydrogen peroxide to terminate the reaction, resulting in a color change to yellowish-brown. The obtained product was washed repeatedly with dilute HCl and deionized water to remove impurities and residual ions. Finally, the material was dried at a moderate temperature to obtain GO powder [11].

2.3. Synthesis of GO-NiO Nanocomposites

GO-NiO nanocomposites were synthesized using a simple co-precipitation method. A calculated amount of GO was dispersed in

deionized water using ultrasonication to achieve a uniform suspension. Nickel nitrate solution was then added to the GO dispersion under continuous stirring. The pH of the solution was adjusted using NaOH to facilitate the formation of nickel hydroxide precursors on the GO surface. The mixture was stirred for several hours to ensure homogeneous interaction between GO and nickel ions. The resulting precipitate was filtered, washed thoroughly with deionized water and ethanol, and dried. The dried material was then calcined at an elevated temperature to convert the precursor into GO-NiO nanocomposites [12].

2.4. Characterization Techniques

The crystalline structure of the synthesized materials was analyzed using X-ray diffraction (XRD). Surface morphology and microstructural features were examined by scanning electron microscopy (SEM). Elemental composition was confirmed using energy-dispersive X-ray spectroscopy (EDS). Functional groups present in the materials were identified using Fourier transform infrared spectroscopy. Optical properties and band gap energy were determined using ultraviolet (UV)-visible spectroscopy. These characterization techniques provided comprehensive information about the structural and physicochemical properties of GO and GO-NiO nanocomposites.

2.5. Photocatalytic Activity Evaluation

The photocatalytic performance of the synthesized materials was evaluated by the degradation of an organic dye under visible light irradiation. A known concentration of dye solution was prepared, and a specific amount of photocatalyst was added. Before light exposure, the suspension was stirred in the dark to establish adsorption-desorption equilibrium. The reaction mixture was then exposed to a

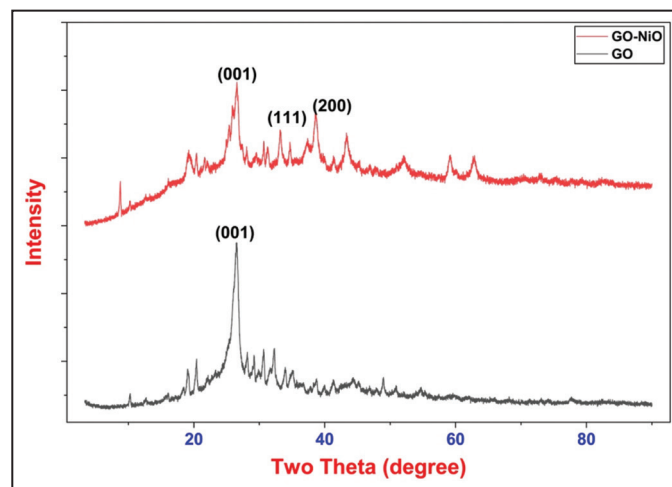


Figure 1: X-ray diffraction of graphene oxide (GO) and GO-Nickel oxide.

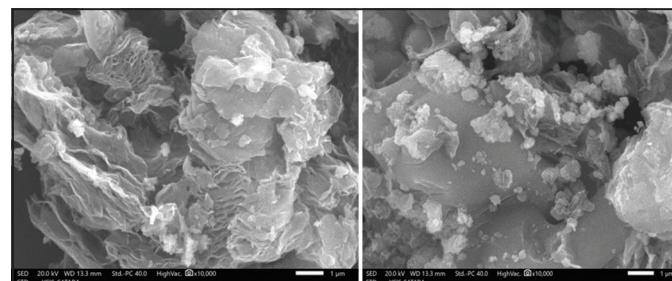


Figure 2: Scanning electron microscopy of graphene oxide (GO) and GO-Nickel oxide.

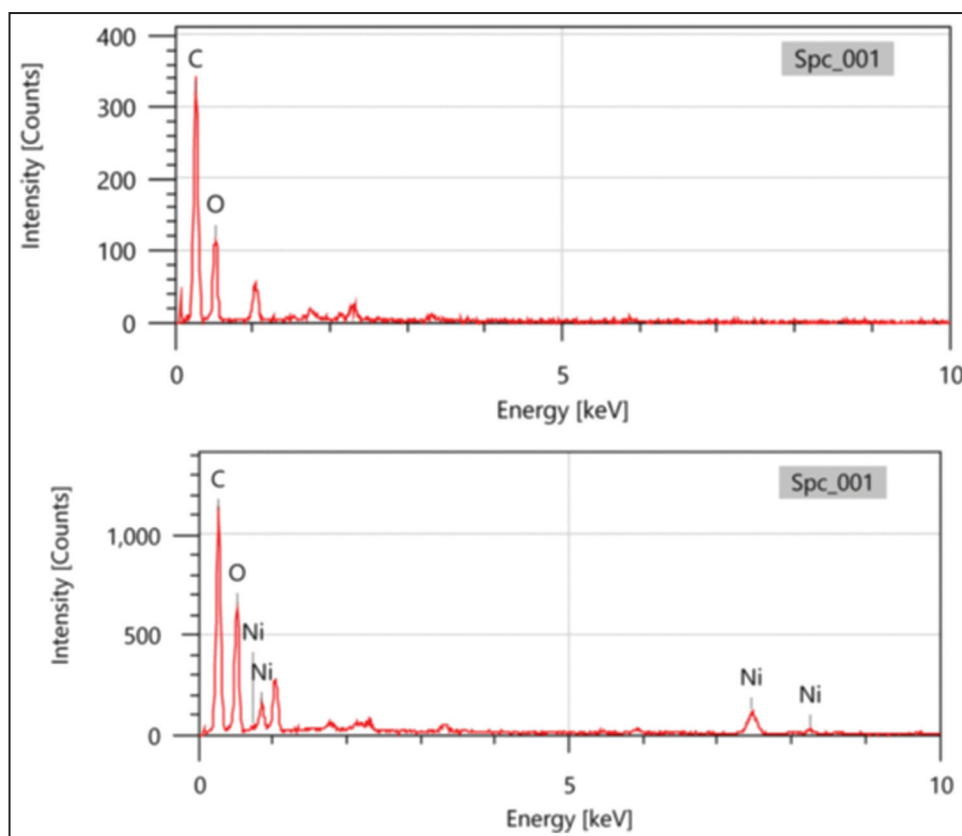


Figure 3: EDS of graphene oxide (GO) and GO-Nickel oxide.

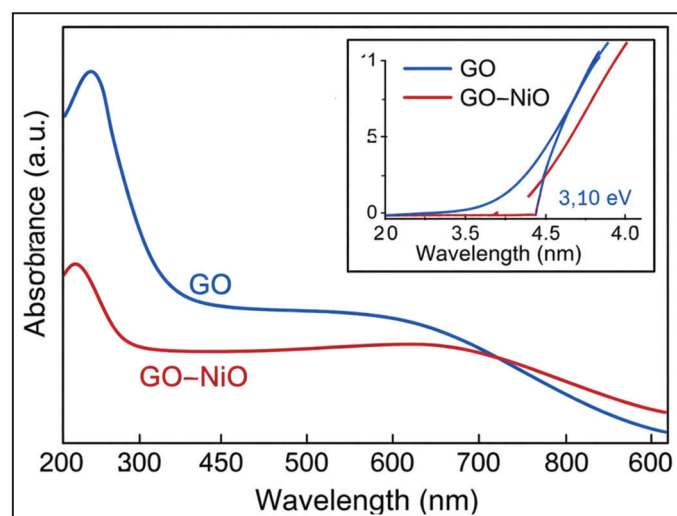


Figure 4: UV-Vis of graphene oxide (GO) and GO-Nickel oxide.

visible light source under continuous stirring. At regular time intervals, aliquots were withdrawn, centrifuged to remove catalyst particles, and analyzed using UV-visible spectroscopy to monitor the decrease in dye concentration. The degradation efficiency was calculated based on the change in absorbance over time.

3. RESULTS AND DISCUSSION

3.1. Structural Analysis (XRD)

The crystalline structure of GO and GO-NiO nanocomposites was examined using XRD analysis [Figure 1]. The XRD pattern of GO

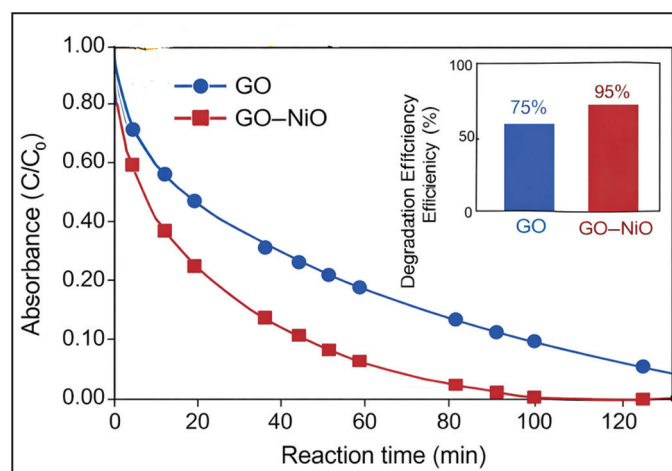


Figure 5: Photocatalytic activity of graphene oxide (GO) and GO-Nickel oxide.

exhibited a characteristic broad diffraction peak around $2\theta \approx 10-12^\circ$, corresponding to the (001) plane, confirming the successful oxidation of graphite and formation of a layered GO structure. In contrast, the GO-NiO nanocomposite showed additional diffraction peaks corresponding to NiO, indicating the successful incorporation of NiO onto the GO matrix. The absence of impurity peaks confirmed the high purity of the synthesized nanocomposite. The average crystallite size of NiO in the composite was calculated using the Debye-Scherrer equation, suggesting the formation of nanocrystalline particles that are well distributed on GO sheets [13].

3.2. Morphological Analysis (SEM)

SEM images revealed significant differences in surface morphology between GO and GO-NiO samples [Figure 2]. The GO sample

Table 1 : Element detection of GO.

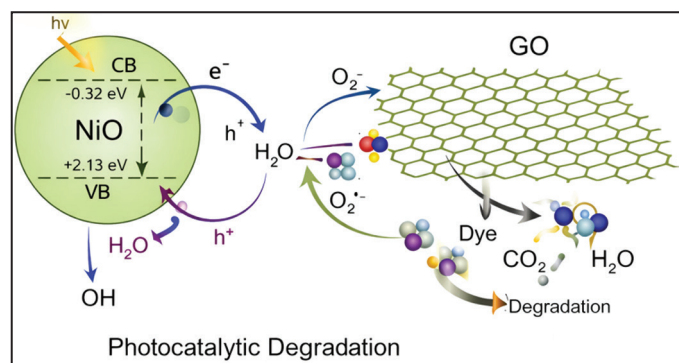
Element	Symbol	X-ray series	Atomic percentage	Weight percentage
Carbon	C	K α -series	63.64	69.12
Oxygen	O	K α -series	36.36	30.88
		Total	100	100

GO: Graphene oxide

Table 2: Element detection of GO-NiO composite.

Element	Symbol	X-ray series	Atomic percentage	Weight percentage
Carbon	C	K α -series	55.00	32.5
Oxygen	O	K α -series	25.00	24.2
Nickel	Ni	K α -series	20.00	43.3
		Total	100	100

GO-NiO: Graphene oxide–Nickel oxide

**Figure 6:** Mechanism of Photocatalytic activity of graphene oxide (GO) and GO-Nickel oxide.

exhibited a typical wrinkled and layered sheet-like structure, which provides a high surface area for adsorption. In the case of GO-NiO nanocomposites, NiO nanoparticles were observed to be uniformly distributed over the GO sheets. This uniform dispersion prevents agglomeration and increases the number of active sites available for photocatalytic reactions. The porous and heterogeneous surface structure further enhances the adsorption of dye molecules, thereby improving photocatalytic efficiency [10].

3.3. Elemental Composition (EDS)

Energy-dispersive X-ray spectroscopy confirmed the presence of carbon and oxygen elements in GO, verifying its composition [Figure 3, Tables 1 and 2]. For the GO-NiO nanocomposite, additional peaks corresponding to nickel were observed along with carbon and oxygen, confirming the successful formation of the composite material as depicted in the figure and tables. The absence of unwanted elemental signals indicates that the synthesized materials are free from significant impurities [9].

3.4. Optical Properties (UV-Vis Analysis)

The UV-Visible absorption spectra of GO and GO-NiO nanocomposites reveal significant differences in their optical behavior [Figure 4]. The GO sample exhibits a strong absorption peak around ~230 nm, which is attributed to the $\pi \rightarrow \pi^*$ transition of aromatic C=C bonds present in the sp^2 hybridized carbon network. In addition, a weak shoulder observed

near ~300 nm corresponds to $n \rightarrow \pi^*$ transitions of C=O functional groups, confirming the presence of oxygen-containing functionalities in GO. In contrast, the GO-NiO nanocomposite shows a noticeable shift in absorption toward the visible region. The absorption edge is extended up to ~350–420 nm, indicating enhanced light-harvesting capability. This red shift suggests strong interaction between GO sheets and NiO nanoparticles, which modifies the electronic structure of the composite material. The band gap energy, estimated using the Tauc method, decreases from approximately 3.10 eV for GO to ~2.45 eV for GO-NiO nanocomposites. This reduction in band gap is a crucial factor contributing to improved visible light absorption and photocatalytic activity. The enhanced optical absorption of GO-NiO can be attributed to effective charge transfer between NiO and GO. NiO acts as a semiconductor that generates electron-hole pairs under light irradiation, while GO serves as an electron acceptor and transport medium, reducing recombination losses. Overall, the UV-Vis analysis confirms that the incorporation of NiO into GO significantly improves optical properties, making GO-NiO nanocomposites more efficient photocatalysts under visible light irradiation [14].

3.5. Photocatalytic Activity

The photocatalytic performance of GO and GO-NiO nanocomposites was evaluated through the degradation of an organic dye under visible light irradiation [Figure 5]. The results demonstrated that the GO-NiO nanocomposite exhibited significantly higher degradation efficiency compared to pure GO. This enhanced performance can be attributed to the synergistic interaction between GO and NiO. GO provides a large surface area for the adsorption of dye molecules, while NiO acts as an active semiconductor that generates electron-hole pairs under light irradiation. The improved photocatalytic efficiency of GO-NiO is primarily due to effective charge separation. Electrons generated in NiO are transferred to GO, which acts as an electron acceptor, thereby reducing recombination of electron-hole pairs. This process enhances the formation of reactive oxygen species such as hydroxyl radicals and superoxide ions, which play a key role in the degradation of dye molecules [15].

3.6. Mechanism of Photocatalytic Degradation

Under visible light irradiation, NiO absorbs photons and generates electron-hole pairs [Figure 6]. The photogenerated electrons are transferred to the GO sheets due to their excellent electron mobility, while the holes remain in NiO. This spatial separation suppresses recombination and prolongs the lifetime of charge carriers. The transferred electrons react with dissolved oxygen to produce superoxide radicals, whereas holes react with water molecules to generate hydroxyl radicals. These highly reactive species attack and degrade the dye molecules into simpler, non-toxic compounds such as CO_2 and H_2O . Give me an image from these data [7].

4. CONCLUSION

In this study, GO and GO-NiO nanocomposites were successfully synthesized and comparatively evaluated for their photocatalytic performance toward dye degradation under visible light irradiation. Structural analysis confirmed the successful formation of GO with a characteristic layered structure, while the incorporation of NiO nanoparticles into the GO matrix was verified through distinct diffraction peaks without any detectable impurities. Morphological observations revealed that NiO nanoparticles were uniformly distributed over the GO sheets, providing a porous and high surface area structure that is favorable for catalytic activity. Elemental analysis further validated the presence of carbon, oxygen, and nickel, confirming the successful synthesis of the nanocomposite. Optical studies demonstrated that

the GO-NiO nanocomposite exhibited enhanced absorption in the visible region along with a reduced band gap compared to pure GO, indicating improved light-harvesting capability. This modification plays a crucial role in enhancing photocatalytic efficiency under visible light conditions. The photocatalytic experiments clearly showed that GO-NiO nanocomposites possess significantly higher degradation efficiency than pure GO, highlighting the advantage of composite formation. The improved performance of GO-NiO can be attributed to the synergistic interaction between GO and NiO, which facilitates efficient charge separation and suppresses electron-hole recombination. GO acts as an electron acceptor and conductive support, whereas NiO provides active sites for redox reactions, leading to enhanced generation of reactive oxygen species responsible for dye degradation. In addition, the large surface area of GO promotes greater adsorption of dye molecules, further contributing to the overall efficiency. Overall, the findings demonstrate that GO-NiO nanocomposites are promising, cost-effective, and efficient photocatalysts for environmental remediation applications. This study provides valuable insights into the design of advanced nanocomposite materials for sustainable wastewater treatment and highlights their potential for large-scale practical implementation.

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*Bibliographical Sketch



Mr. Sandesh V. Gaikwad is currently working as Ph.D. scholar in MG V.M.S.G. College, Malegaon. He received B.Sc. and M.Sc. degree in chemistry from Savitribai Phule Pune University, Pune. The corresponding author is a dedicated researcher in the field of chemical sciences, specializing in nanomaterials and their environmental applications. Their research focuses on the synthesis, characterization, and application of graphene oxide and transition metal oxide-based nanocomposites for photocatalysis and gas sensing. The author has been actively involved in research aimed at addressing environmental pollution through innovative material design. They have contributed to academic publications in peer-reviewed journals and regularly participate in scientific conferences and workshops.