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BIOMASS-DERIVED REDUCED GRAPHENE OXIDE FOR EFFICIENT PHOTOCATALYTIC DEGRADATION OF METHYL ORANGE DYE

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Abstract

This study addresses environmental concerns related to water pollution caused by persistent and toxic azo dyes such as methyl orange. A sustainable approach is proposed using biomass-derived reduced graphene oxide (rGO) synthesized through a green reduction method employing renewable plant or agricultural waste extracts. The prepared rGO is characterized using XRD, and Raman spectroscopy to confirm structural, morphological, and optical properties. The photocatalytic efficiency of the material is evaluated through the degradation of methyl orange under light irradiation. Key operational parameters including catalyst dosage, dye concentration, pH, and irradiation time are systematically optimized. Results are expected to demonstrate enhanced photocatalytic activity due to improved surface area and reduced charge recombination. Ultimately, the study aims to develop an eco-friendly, cost-effective photocatalyst capable of efficient mineralization of pollutants into harmless products like CO_2 and H_2O.

Keywords: Biomass-derived reduced graphene oxide (rGO), Green synthesis, Photocatalytic degradation, Methyl orange dye

1. Introduction

Water pollution caused by synthetic dyes has become a serious environmental concern due to rapid industrialization. Among various pollutants, azo dyes such as methyl orange are widely used in textile, paper, printing, and pharmaceutical industries [1-3]. These dyes are highly stable, non-biodegradable, and toxic, causing severe ecological and health problems when released into water bodies. Therefore, developing efficient, eco-friendly, and cost-effective methods for dye degradation is essential [4]. Photocatalysis has emerged as a promising technique for removing organic pollutants from wastewater. Semiconductor photocatalysts generate reactive oxygen species under light irradiation, which break down complex dye molecules into harmless products like carbon dioxide and water. However, conventional photocatalysts suffer from low efficiency due to rapid recombination of electron–hole pairs and limited surface area [5-7]. Reduced graphene oxide has gained attention as an effective material in photocatalytic systems. It possesses excellent electrical conductivity, large surface area, and strong adsorption capacity. It also acts as an electron acceptor, reducing charge recombination and improving photocatalytic efficiency [8-10]. Traditional synthesis methods of graphene materials often involve toxic chemicals, which can harm the environment. Therefore, biomass-derived carbon materials are being explored as sustainable alternatives. Biomass sources such as agricultural waste and plant extracts are renewable, low-cost, and environmentally friendly. These materials can be used for the green synthesis of graphene oxide and its reduced form. Biomass not only reduces environmental impact but also introduces functional groups that enhance photocatalytic performance [11-13]. This study focuses on synthesizing biomass-derived reduced graphene oxide for degrading methyl orange dye. The prepared material is expected to show improved adsorption capacity and enhanced charge separation. It also exhibits superior degradation efficiency under light irradiation. Such materials provide a sustainable solution for wastewater treatment. The synthesis of nanomaterials plays a crucial role in determining their properties such as size, shape, and morphology [14-16]. There are two main synthesis approaches: physical and chemical methods. Chemical methods are preferred due to their simplicity, low cost, and environmental compatibility. Techniques such as sol-gel, hydrothermal, and precipitation methods are commonly used. Among these, the sol-gel method offers advantages like high purity and controlled structure [17-19]. Nanostructured materials show unique optoelectronic properties compared to bulk materials. These properties are studied using various analytical and spectroscopic techniques. The interaction of light with materials helps in understanding their structural and electronic characteristics. The overall properties of nanomaterials depend on synthesis methods and operating conditions. Proper characterization is essential to evaluate their performance in applications. In photovoltaic device fabrication, thin film deposition is a key step. Methods like the doctor blade technique are widely used due to simplicity and low cost. This technique allows easy fabrication of thin films for nanocomposite materials. The performance of such devices is analysed using current-voltage measurements. Overall, the development of biomass-derived rGO offers a promising approach for environmental remediation and advanced material applications.

1.1 Objectives of the Proposed Research

The main objective of this research is to develop a sustainable and efficient photocatalyst using biomass-derived reduced graphene oxide (rGO) for the degradation of methyl orange dye from wastewater. The study focuses on the green synthesis of rGO using renewable biomass materials, avoiding toxic chemical reducing agents. The synthesized material is characterized using techniques such as XRD, Raman spectroscopy to analyse its structural and optical properties. The photocatalytic performance of rGO is evaluated by studying its efficiency in degrading methyl orange under light irradiation, along with calculating degradation percentage and reaction kinetics. The effect of various operational parameters like catalyst dosage, dye concentration, pH, and irradiation time is also examined to optimize conditions. Additionally, the degradation mechanism is investigated by understanding the role of reactive oxygen species and charge separation processes. Finally, the stability and reusability of the photocatalyst are assessed through recycling studies to determine its practical applicability.

1.2 Research Methodology

The research methodology involves a systematic approach to synthesize and evaluate biomass-derived reduced graphene oxide (rGO) for photocatalytic dye degradation. Initially, suitable biomass sources, graphite powder, methyl orange dye, and other chemicals are collected, and distilled water is prepared for experiments [20]. Graphene oxide (GO) is synthesized from graphite using the modified Hummers' method, followed by washing and drying. The green synthesis of rGO is carried out by preparing a biomass extract, mixing it with GO under controlled conditions, and reducing GO using natural phytochemicals, followed by purification and drying. The synthesized rGO is then characterized using techniques such as XRD, and Raman spectroscopy to study its structure and properties. Photocatalytic experiments are performed by adding rGO to methyl orange dye solution, exposing it to light, and measuring absorbance at regular intervals to determine degradation efficiency. Optimization studies are conducted by varying parameters like catalyst dosage, dye concentration, pH, and irradiation time. The degradation kinetics and mechanism are analysed using models and reactive species studies. The reusability and stability of the catalyst are evaluated through repeated cycles. Finally, data is analysed by plotting degradation curves, calculating rate constants, and interpreting the relationship between structure and performance [21].

1.3 Expected Outcomes

The expected outcomes of this research include the successful green synthesis of reduced graphene oxide (rGO) using biomass as a natural and eco-friendly reducing agent, minimizing the use of toxic chemicals. The synthesized rGO is anticipated to exhibit improved structural and surface properties, such as high surface area, enhanced conductivity, and the presence of functional groups that boost photocatalytic activity. The material is expected to show high efficiency in the degradation of methyl orange dye under light irradiation, converting it into harmless products like CO_2 and H_2O at a faster rate than conventional catalysts. Enhanced charge separation and better electron transport are also expected, reducing recombination losses. The study aims to optimize operational parameters such as

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catalyst dosage, pH, dye concentration, and irradiation time for maximum performance. Additionally, the photocatalyst is expected to demonstrate good stability and reusability over multiple cycles with minimal loss of efficiency. Overall, the research will provide a sustainable, cost-effective, and environmentally friendly solution for wastewater treatment and may be applicable for removing other organic pollutants.

2. Materials and methods

2.1 Materials

A suitable biomass source Soyabean Husk was used as a natural reducing agent due to its rich phytochemical content (polyphenols, flavonoids, and sugars), enabling eco-friendly reduction of graphene oxide (GO) to reduced graphene oxide (rGO). Graphite powder served as the precursor for GO synthesis.

Analytical grade chemicals including sulfuric acid (H_2SO_4), potassium permanganate ($KMnO_4$), and hydrogen peroxide (H_2O_2) were used in the modified Hummers' method. Distilled/deionized water was used for all preparations and washing steps.

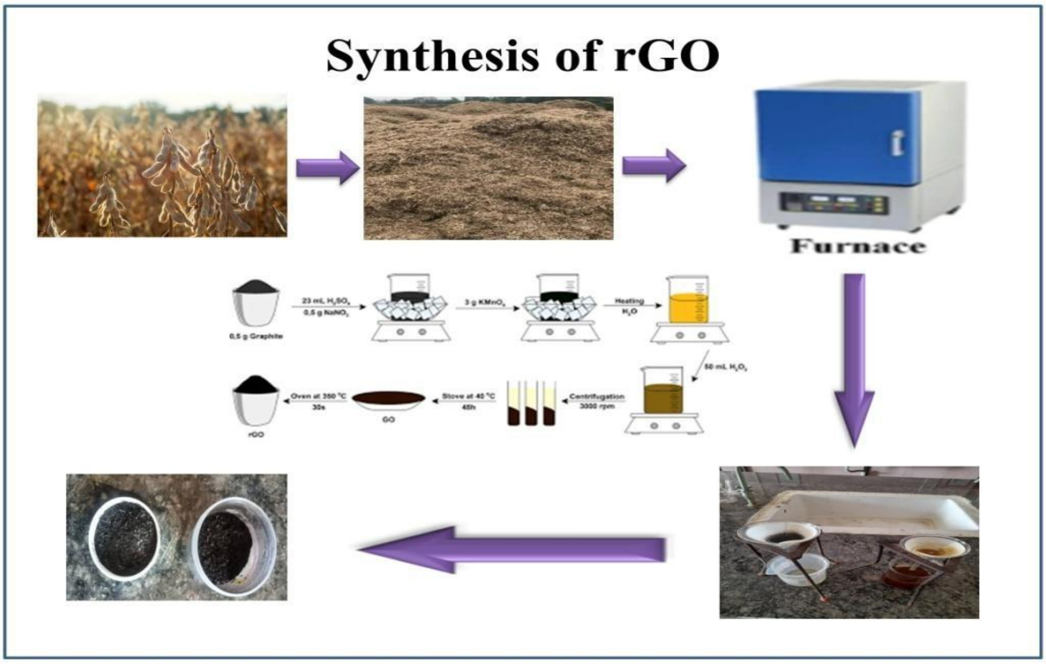
Methyl orange (MO) dye was selected as a model pollutant for photocatalytic degradation studies. The synthesized biomass-derived rGO acted as the photocatalyst due to its high surface area, electrical conductivity, and adsorption capacity.

Ethanol/acetone was used for washing purposes. Standard laboratory glassware and filter paper were used for experimental procedures. A UV/visible light source was employed to initiate photocatalytic reactions.

2.2 Synthesis method and Characterization Techniques of Nanomaterials

2.2.1 Synthesis

The synthesis of nanomaterials plays a crucial role in determining their physical and chemical properties such as size, shape, morphology, and crystallinity. Based on literature studies, nanomaterials can be synthesized using two major approaches: physical methods and chemical methods. Among these, chemical methods are more widely used due to their simplicity, cost-effectiveness, and environmentally friendly nature. Various synthesis routes such as thermal decomposition, sol-gel, sonochemical, hydrothermal, and co-precipitation methods are commonly employed for the preparation of nanoparticles, including reduced graphene oxide (rGO).



Schematic representation of synthesis of nanoparticles of reduced graphene oxide.

2.2.2 Characterization Techniques

Characterization of nanomaterials is essential to understand their structural, morphological, and chemical properties. Various analytical tools are used to study these properties, which are important for determining their performance in applications such as photocatalysis.

X-ray Diffraction (XRD) Analysis

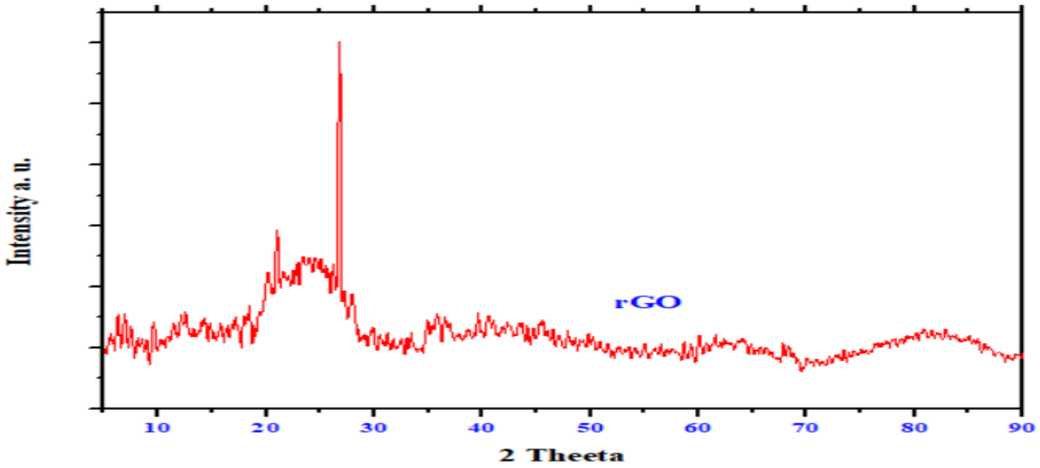
XRD is a powerful technique used to determine the crystal structure, phase composition, and interplanar spacing of materials. It works based on the diffraction of X-rays when they interact with atomic planes in a crystal. The diffraction pattern provides information about the crystallinity and structure of the material. The interplanar spacing is calculated using Bragg's law, which relates the wavelength of X-rays to the diffraction angle.

Raman Spectroscopy

Raman spectroscopy is used to study molecular vibrations and structural properties of materials. It is based on the Raman effect, where light scattering causes a shift in energy due to molecular interactions. This technique is especially useful for analysing carbon-based materials such as graphene and reduced graphene oxide. It provides information about defects, disorder, and graphitic structure. Raman spectroscopy is a non-destructive method and requires minimal sample preparation.

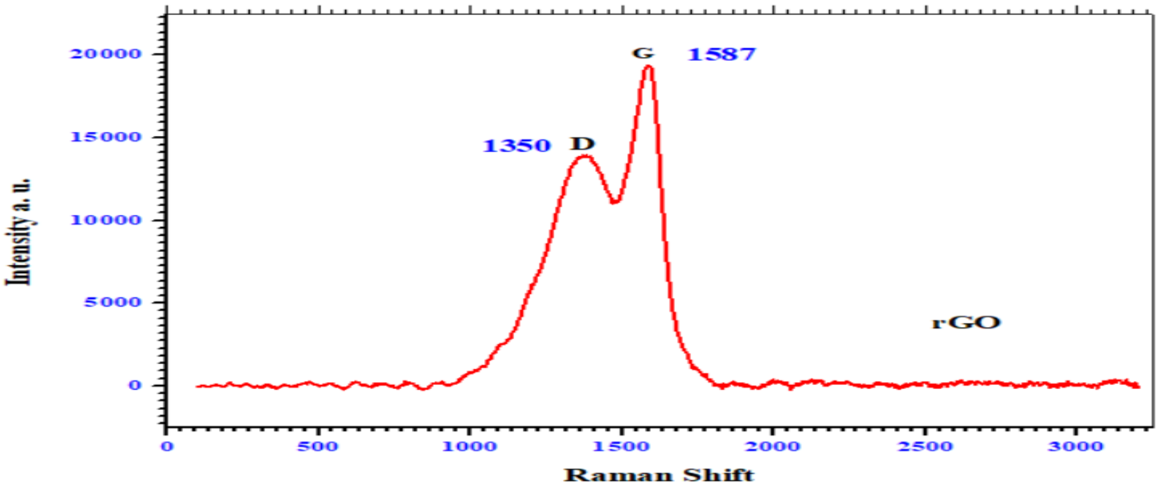
3. Result and Discussion

3.1 XRD Analysis of rGO



The XRD pattern of reduced graphene oxide (rGO) shows a broad peak at 2θ ≈ 24–26°, indicating the restoration of graphitic structure after reduction. Compared to graphene oxide (GO), which shows a peak at 10–12°, the shift confirms the removal of oxygen-containing groups and reduced interlayer spacing. The broad and low-intensity peak suggests poor crystallinity and a disordered structure. Overall, the XRD results confirm the successful conversion of GO into rGO.

3.2 Raman Spectroscopy of rGO



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The Raman spectrum of rGO shows two main peaks: the D band (~1350 cm^{-1}) and G band (~1587 cm^{-1}). The G band indicates graphitic structure, while the D band represents defects and disorder. A higher D/G intensity ratio suggests more structural defects. The presence of both bands confirms the successful reduction of GO to rGO with a partially restored structure. This defective but conductive structure makes rGO suitable for photocatalytic applications.

3.3 Application for the decomposition of toxic methyl orange dye

Synthetic dyes are extensively used in textile and leather industries, and their demand is increasing with industrial growth. These dyes are produced from harmful chemicals such as aromatic hydrocarbons and heterocyclic compounds, which pose serious environmental and health risks. During dyeing processes, a significant amount of dye (about 5–10%) is released into wastewater, leading to contamination of water bodies like rivers, lakes, and oceans. This pollution adversely affects aquatic life and disrupts ecosystems. [22-24]

Among various dyes, azo dyes constitute nearly 70% and are widely used due to their stability and colour properties. However, their degradation produces toxic aromatic amines, which are carcinogenic and can accumulate in human organs such as the liver, kidney, and bladder. Industrial discharge of such dye-containing wastewater from textile, leather, and printing industries has become a major environmental concern.[25]

Various conventional methods such as sedimentation, filtration, coagulation, oxidation, and biodegradation have been used to treat dye wastewater. However, these methods are often inefficient as they only transfer pollutants from one phase to another and may create secondary pollution. Chemical treatments can remove colour but fail to completely degrade harmful compounds, sometimes producing more toxic byproducts.[26]

Dyes are classified into natural and synthetic types and further into categories such as acidic, basic, mordant, vat, and azo dyes. When released untreated, they cause toxicity, mutagenicity, and serious environmental damage.[27] Therefore, effective degradation of these dyes before discharge is essential.

Photocatalysis has emerged as an advanced and efficient method for dye degradation. It involves the use of photocatalysts like rGO under UV or visible light to break down dye molecules into harmless substances. Methyl orange, a commonly used azo dye, is widely applied in textile dyeing and as an indicator in chemical analysis.[28]

In this study, nanomaterials were used as photocatalysts in a photocatalytic reactor for the degradation of methyl orange dye [29]. Important parameters such as contact time and catalyst amount were optimized, and degradation was monitored at a wavelength of 464 nm. The results showed high degradation efficiency, indicating that the synthesized nanomaterials are effective for treating dye-contaminated wastewater under optimal conditions.[30]

Reduced graphene oxide photodegradation

The effects of time, catalyst quantity, percentage degradation, and catalyst reusability for methyl orange dye were investigated

3.3.1 Time's Impact

Methyl orange was utilized in this investigation in an aqueous solution at a concentration of 1x10-3 M. Before being exposed to radiation, a 10 cm^3 sample of this dye solution and 0.100 g of produced copper oxide catalyst were magnetically agitated for 30 minutes in the dark to achieve adsorption-desorption equilibrium between the dye and the catalyst surface. Over a period of 0 to 240 minutes, the dye's degradation was observed. The ideal degradation percentage (% D) was found to be reached between 210 and 240 minutes. The results are presented in Figures 3.1 to and summarized in Tables.

Table 3.1: Effect of time on methyl orange degradation with Reduced graphene oxide

Time (min)	**Absorbance**	**% X**	**% D**
0	0.748	100	0

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15	0.748	100	0
30	0.748	100	0
45	0.626	83.68	16.32
60	0.603	80.61	19.39
75	0.585	78.20	21.08
90	0.545	72.86	27.14
105	0.520	69.51	30.49
120	0.491	65.64	34.36
135	0.340	45.45	54.55
150	0.325	43.44	56.56
165	0.225	30.08	69.92
180	0.151	20.18	79.82
195	0.081	10.82	89.18
210	0.054	7.21	92.79
225	0.054	7.21	92.79
240	0.054	7.21	92.79

Figure-3.1: Effect of time on methyl orange degradation with Reduced graphene oxide

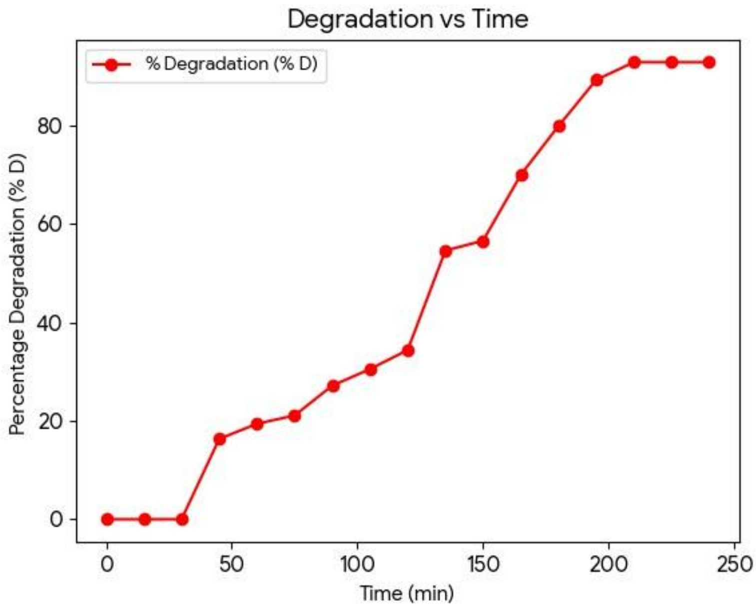

Figure-3.2: Effect of time on MO degradation with Reduced graphene oxide (% D Values)

3.3.2 Effect of the amount of catalyst

Using 10 cm3 of dye solution and varying rGO catalyst quantities in the reaction, the impact of catalyst quantity was examined. By evaluating several amounts ranging from 0.025g to 0.150g and carrying out the photodegradation of methyl orange dye for 240 minutes, the ideal quantity of photocatalyst was determined. The samples were measured for absorbance at a wavelength of 464 nm. At 0.100g of catalyst, the maximum degradation was achieved. The results are shown in Figure below and summarized in Table 3.2.

Table-3.2: Effect of amount of catalyst on methyl orange dye degradation

Amount of catalyst in g	Absorbance	% X	% D
0.025	0.185	20.21	79.79
0.050	0.126	13.76	86.24
0.075	0.081	8.85	91.15
0.100	0.054	7.21	92.79
0.125	0.054	7.21	92.79
0.150	0.054	7.21	92.79
0.175	0.054	7.21	92.79
0.200	0.054	7.21	92.79

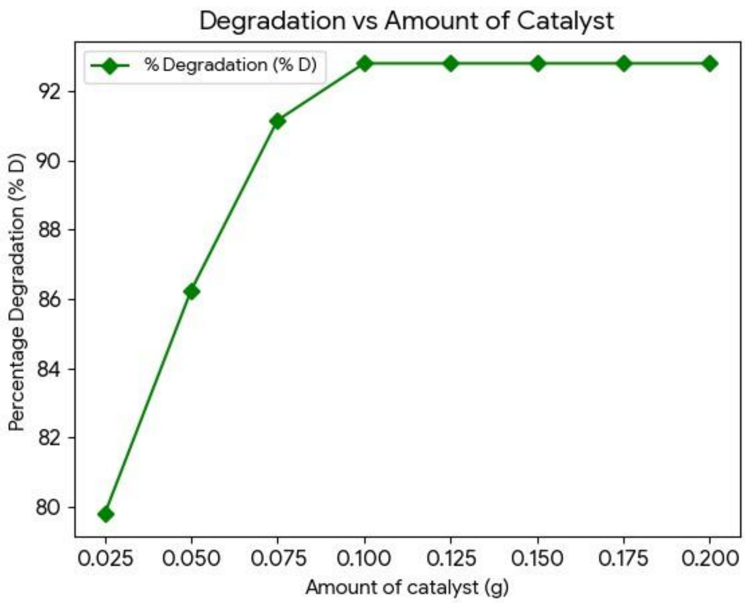

Figure-3.3: Effect of amount of catalyst of reduced graphene oxide to methyl orange dye degradation

3.3 3 Reusability of catalyst

Centrifugation and filtering were used to extract the catalysts from the degraded dye solution. They were then successively cleaned with distilled water and acetone before being dried in an oven. The rGO nanomaterial catalyst was evaluated over multiple catalytic cycles to evaluate its lifetime and

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reusability. As shown in Table , it was discovered that the reduced graphene oxide produced from hydrazone ligand copper complexes continued to be effective for four cycles before experiencing a minor drop-in catalytic activity.

Table 3.3: Reusability of catalyst

No. of catalytic cycles	Absorbance	% X	% D
1	0.054	7.21	92.79
2	0.054	7.21	92.79
3	0.054	7.21	92.79
4	0.072	8.42	91.58
5	0.086	9.14	90.86
6	0.097	9.75	90.25

4. Conclusion

- ✓ The present study successfully demonstrated the green synthesis of reduced graphene oxide (rGO) using biomass as a sustainable and eco-friendly precursor, eliminating the use of toxic chemical reducing agents.
- ✓ The synthesized rGO exhibited improved structural and surface properties, as confirmed by XRD and Raman analysis, indicating partial restoration of graphitic structure along with the presence of defects that enhance catalytic activity.
- ✓ The prepared material showed excellent photocatalytic performance in the degradation of methyl orange dye, achieving a maximum degradation efficiency of about 92.79% under optimized conditions.
- ✓ The study revealed that parameters such as reaction time and catalyst dosage significantly influence the degradation efficiency, with optimum performance observed at 0.100 g catalyst and 210–240 minutes of irradiation.
- ✓ The enhanced photocatalytic activity is mainly attributed to efficient electron–hole separation, high surface area, and strong adsorption capacity of rGO, which facilitates the generation of reactive oxygen species responsible for dye degradation.
- ✓ The degradation process followed pseudo-first-order kinetics, confirming the efficiency of the photocatalyst. Furthermore, the catalyst exhibited good stability and reusability over multiple cycles with only a slight decrease in performance.

Overall, the study highlights that biomass-derived rGO is a cost-effective, sustainable, and efficient material for wastewater treatment, offering a promising approach for environmental remediation and supporting green nanotechnology advancements.

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