

## Synthesis And Characterization Applications Of Nanoparticles For Photocatalytic Degradation Of Organic Dyes

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### Abstract

*Nanoparticle-enabled photocatalysis offers a low-energy, reagent-lean route for degrading persistent organic dyes in wastewater. This paper surveys the synthesis–structure–function nexus across metal oxides ( $TiO_2$ ,  $ZnO$ ,  $Fe_3O_4$ ), sulfides (CoS, CdS-based), carbon nitride ( $g-C_3N_4$ ), and conductive polymer and MOF-derived hybrids engineered for visible-light response. Synthetic routes—including sol–gel, hydro/solvothermal, microemulsion, combustion, and green biogenic methods—are mapped to crystallinity, defect chemistry, and facet exposure that tune band gaps and charge transport. Comprehensive characterization (XRD, Raman, FTIR, XPS, TEM/HRTEM, SEM/EDS, UV–Vis DRS, PL/TRPL, BET, zeta potential) is used to correlate heterojunction architectures (S, Z, direct Z-scheme), dopant states (V, Ni/Fe, non-metals), and cocatalysts with reactive oxygen species generation ( $\bullet OH$ ,  $\bullet O_2^-$ ,  $^1O_2$ ). Kinetic behavior typically follows pseudo-first-order models, with performance reported for dyes such as methylene blue, rhodamine B, methyl orange, and Congo red under simulated solar irradiation. Stability ( $\geq 5$  reuse cycles), mineralization (TOC removal), and anti-fouling behavior are discussed alongside toxicity and leachate controls. Finally, we outline scalable reactor designs (fixed-film coatings, magnetic recovery, membrane-coupled systems) and reporting standards (spectral photon flux, space-time yield, apparent quantum efficiency) needed to bridge lab efficacy with real effluent treatment.*

**Keywords:** photocatalysis; nanoparticles; dye degradation; heterojunctions; bandgap engineering; green synthesis

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### INTRODUCTION

The growing global demand for water resources has been severely challenged by the alarming rise in environmental pollution, especially in the context of industrial effluents containing toxic and non-biodegradable contaminants. Among various pollutants, organic dyes used extensively in textile, leather, cosmetics, and paper industries represent one of the most persistent categories of chemical waste discharged into water bodies. Dyes such as methylene blue, rhodamine B, methyl orange, and Congo red are not only aesthetically undesirable due to coloration of water but also pose carcinogenic, mutagenic, and cytotoxic threats to aquatic life and human health. Their structural stability, aromatic frameworks, and synthetic complexity make them resistant to natural degradation and conventional treatment methods such as coagulation, sedimentation, and biological degradation. As a consequence, the search for efficient, sustainable, and scalable remediation technologies has intensified in recent years, with photocatalysis emerging as one of the most promising approaches.

Photocatalytic degradation leverages semiconductor nanomaterials that, upon exposure to suitable irradiation, generate electron-hole pairs capable of initiating a cascade of redox reactions. These reactions lead to the production of highly reactive oxygen species (ROS), such as hydroxyl radicals ( $\bullet OH$ ), superoxide anions ( $\bullet O_2^-$ ), and singlet oxygen ( $^1O_2$ ), which can attack and mineralize organic dye molecules into harmless end-products like  $CO_2$  and  $H_2O$ . The field has advanced substantially through the integration of nanotechnology, enabling the synthesis of nanoparticles with tailored morphologies,

controlled surface states, defect engineering, and heterojunction formation. These features enhance light absorption, facilitate charge separation, and suppress recombination, thereby improving photocatalytic efficiency. Furthermore, the incorporation of green synthesis strategies, doping with metals and non-metals, and hybridization with carbon-based nanomaterials or metal-organic frameworks (MOFs) has added a new dimension to the design of photocatalysts aimed at real-world wastewater treatment.

### Overview

The present research paper reviews and discusses in depth the synthesis strategies and characterization techniques of nanoparticles that have been applied toward photocatalytic degradation of organic dyes. While  $\text{TiO}_2$  and  $\text{ZnO}$  remain the most studied metal oxides, considerable attention has shifted to transition metal sulfides, ferrites, doped composites, carbon nitride-based semiconductors, and polymer-supported or MOF-derived nanostructures. Each class of material exhibits unique advantages such as extended absorption in the visible range, improved charge carrier dynamics, or superior stability. However, practical application requires a holistic understanding of how the synthesis method influences nanoparticle features such as crystallinity, particle size, surface chemistry, and defect density, which in turn govern photocatalytic outcomes. To provide this understanding, characterization techniques including X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy, scanning and transmission electron microscopy (SEM, TEM), photoluminescence (PL), UV-Vis diffuse reflectance spectroscopy (DRS), Brunauer-Emmett-Teller (BET) surface area analysis, and X-ray photoelectron spectroscopy (XPS) are evaluated for their roles in establishing structure-property-performance relationships.

### Scope and Objectives

The scope of this study is twofold: (i) to consolidate the latest research advances in the synthesis and characterization of nanoparticles tailored for photocatalytic degradation of dyes, and (ii) to contextualize these findings within the framework of environmental sustainability and industrial applicability. The specific objectives can be summarized as follows:

1. To critically examine various nanoparticle synthesis routes—conventional (sol-gel, hydrothermal, microemulsion, combustion) and emerging (biogenic, polymer-assisted, template-free)—and analyze their impact on morphology and functionality.
2. To map the role of comprehensive physicochemical characterization in correlating synthesis parameters with photocatalytic efficiency.
3. To highlight the photocatalytic performance of nanoparticles against benchmark dyes under visible and solar irradiation, identifying trends in activity, kinetics, stability, and recyclability.
4. To assess the challenges related to scale-up, toxicity, secondary pollution, and energy economics that hinder industrial deployment of nanoparticle-based photocatalysts.
5. To provide a knowledge-driven roadmap for designing next-generation nanostructured photocatalysts that are environmentally benign, cost-effective, and scalable.

### Author Motivations

The motivation for this work stems from the critical environmental challenges associated with untreated dye effluents in rapidly industrializing economies. The authors recognize that despite significant research efforts, there remains a gap between laboratory-scale studies reporting high degradation efficiencies and their successful translation into industrial wastewater treatment systems. Many studies often lack standardized reporting of photon flux, quantum yields, or stability over repeated cycles, making it difficult to compare results across systems. Furthermore, issues of photocatalyst recovery, reusability, and toxicity assessment remain underexplored. Against this backdrop, the authors aim to synthesize and characterize nanoparticles with an emphasis on reproducibility, comprehensive performance analysis, and alignment with sustainable water management practices. This work is also motivated by the desire to bridge fundamental material science with applied environmental engineering, ultimately fostering technology readiness for real-world adoption.

**Paper Structure**

To present the research in a systematic manner, the paper is structured as follows: Section 1 introduces the background, rationale, scope, and motivations of the study. Section 2 provides a detailed review of existing literature on nanoparticle-based photocatalysts, including metal oxides, metal sulfides, carbonaceous composites, and hybrid systems, with critical emphasis on their mechanisms and performance metrics. Section 3 elaborates on the synthesis methodologies employed for nanoparticle preparation, discussing the influence of synthesis conditions on size, morphology, crystallinity, and surface properties. Section 4 describes the various characterization techniques applied to assess structural, optical, electronic, and surface properties of the synthesized nanoparticles. Section 5 presents the photocatalytic performance evaluation against selected dyes, including kinetic modeling, stability testing, and mechanistic insights. Finally, Section 6 concludes the paper with a summary of findings, implications for sustainable wastewater treatment, and policy-level recommendations.

Overall, the introduction establishes the urgent need for innovative nanoparticle-based solutions to combat organic dye pollution in water systems. It emphasizes the scientific and societal motivations that underlie the present research, clarifies the scope and objectives, and positions the study within the broader discourse on green nanotechnology and environmental remediation. By addressing both the fundamental aspects of nanoparticle synthesis and characterization as well as the applied aspects of photocatalytic degradation, the paper aims to provide a holistic contribution that not only advances academic knowledge but also holds promise for practical implementation in sustainable wastewater treatment.

**2. LITERATURE REVIEW****2.1 Nanoparticle-Based Photocatalysis for Dye Degradation**

The field of photocatalytic degradation of organic dyes has witnessed significant advancements in the last decade, with nanoparticles serving as the cornerstone of material innovation. Classical photocatalysts, such as  $\text{TiO}_2$  and  $\text{ZnO}$ , were initially favored for their wide band gap, photostability, and abundance. However, their limited visible-light activity and rapid electron–hole recombination restricted large-scale applicability. Consequently, research shifted toward bandgap engineering, surface modification, heterojunction formation, and the development of hybrid nanostructures that could enhance photocatalytic efficiency under solar or visible-light irradiation.

Recent studies underscore that the choice of nanomaterials and synthesis routes directly influences the photocatalytic behavior. For instance, vanadium-doped  $\text{TiO}_2$  nanoparticles exhibited superior visible-light activity compared to pristine  $\text{TiO}_2$ , highlighting the role of dopants in narrowing the bandgap and improving charge carrier separation [4]. Similarly,  $\text{ZnO}$ -based photocatalysts continue to attract attention due to their eco-friendliness, facile synthesis, and strong oxidative power. Comprehensive reviews confirm that  $\text{ZnO}$  nanoparticles, when modified with heteroatoms or coupled with secondary semiconductors, can overcome their intrinsic photoinstability and expand their absorption spectrum [6], [10].

**2.2 Metal Oxide Nanoparticles**

Metal oxide nanoparticles remain the most extensively investigated category for photocatalytic dye degradation. Sharma et al. [4] demonstrated that V-doped  $\text{TiO}_2$  nanoparticles achieved improved degradation efficiency for methylene blue and rhodamine B, establishing doping as a viable pathway for activity enhancement. Similarly,  $\text{ZnFe}_2\text{O}_4@\text{Co/Ni-MOF}$  nanocomposites synthesized by Zhang et al. [11] revealed synergistic effects between spinel ferrites and MOF matrices, enhancing charge transfer and extending visible-light absorption.

Hybrid approaches have also proven effective. Al-Zuhairi et al. [13] reported that  $\text{ZnO-TiO}_2-\text{Fe}_3\text{O}_4$  composites achieved robust activity for dye degradation, combining the stability of  $\text{TiO}_2$ , the high reactivity of  $\text{ZnO}$ , and the magnetic recovery ability of  $\text{Fe}_3\text{O}_4$ . This hybridization not only improved photocatalytic performance but also facilitated post-treatment recovery, an essential criterion for practical wastewater applications. Similarly, Khan et al. [14] synthesized  $\text{ZnO-TiO}_2-\text{rGO}$  composites, achieving enhanced dye degradation due to improved charge mobility provided by reduced graphene oxide sheets.

### 2.3 Metal Sulfide and Ferrite-Based Nanoparticles

Beyond metal oxides, transition metal sulfides have gained momentum for visible-light-driven photocatalysis. CoS nanoparticles synthesized through green methods exhibited rapid methylene blue degradation, with mechanistic insights indicating efficient ROS generation due to their narrow band gap [1]. Similarly, ternary ZnxCdS/ZnO/g-C<sub>3</sub>N<sub>4</sub> composites demonstrated exceptional performance under sunlight due to Z-scheme charge transfer mechanisms, which ensured strong oxidation and reduction potentials [8].

Magnetic ferrites have also been extensively studied for their recyclability and visible-light responsiveness. Kamaruddin et al. [2] synthesized NiFe<sub>2</sub>O<sub>4</sub>/g-C<sub>3</sub>N<sub>4</sub> nanocomposites, reporting high activity for rhodamine B degradation due to synergistic effects of NiFe<sub>2</sub>O<sub>4</sub>'s magnetism and g-C<sub>3</sub>N<sub>4</sub>'s visible-light absorption. Such materials not only degrade dyes efficiently but also facilitate catalyst recovery through external magnetic fields, a crucial advantage for industrial applications.

### 2.4 Carbon-Based and Polymer-Assisted Photocatalysts

Carbonaceous materials such as g-C<sub>3</sub>N<sub>4</sub> and graphene-based structures have revolutionized photocatalysis by serving as co-catalysts, electron mediators, and photosensitizers. ZnxCdS/ZnO/g-C<sub>3</sub>N<sub>4</sub> composites exemplify how carbon nitride can act as an electron mediator in heterojunction systems [8]. Additionally, the incorporation of polyaniline nanoparticles has shown promise in enhancing photocatalytic activity under simulated solar light by improving charge transport and increasing visible-light absorption [5]. Such conductive polymers not only enhance photocatalytic activity but also introduce functional groups that contribute to surface adsorption of dyes.

### 2.5 Metal-Organic Framework (MOF)-Derived Nanostructures

Recent studies emphasize the utility of MOF-derived and MOF-integrated systems in dye degradation. Li et al. [3] designed MOF-anchored dibenzo-18-crown-6@polydopamine functionalized membranes that demonstrated dual-functionality—adsorption and photocatalysis—for Congo red removal. Likewise, Liao et al. [12] reviewed MOF-based nanocomposite photocatalysts, highlighting their tunable porosity, large surface area, and hybridization capabilities that offer enhanced activity and selectivity.

MOF-metal oxide composites have further enriched this domain. For instance, ZnFe<sub>2</sub>O<sub>4</sub>@Co/Ni-MOF nanostructures not only enhanced dye degradation but also improved photocatalyst stability and recyclability [11]. These studies highlight the evolving role of MOFs as both scaffolds and active participants in photocatalytic degradation systems.

### 2.6 Emerging Green Synthesis Routes

In parallel to performance-driven studies, the sustainability of nanoparticle synthesis has gained recognition. Singh et al. [9] employed green solvents to prepare ZnO thin films, reporting high activity against organic dyes while minimizing the environmental footprint. Dang et al. [1] and Kamaruddin et al. [2] further employed eco-friendly routes, emphasizing the dual need for effective photocatalysis and environmentally benign synthesis. The trend indicates a shift from energy-intensive, hazardous synthesis methods toward greener, biogenic, and resource-efficient alternatives.

### 2.7 Performance Metrics, Kinetics, and Stability

Across studies, dye degradation kinetics commonly follow pseudo-first-order models, with rate constants increasing upon heterojunction formation or dopant addition. For example, TiO<sub>2</sub> modified with dopants such as V [4] and ZnO-rGO systems [14] demonstrated enhanced kinetics. Etim et al. [10] highlighted how optoelectronic properties of spray-pyrolyzed ZnO thin films influenced kinetic performance. Notably, stability and recyclability remain crucial evaluation criteria. Several studies, including [2], [11], and [13], demonstrated recyclability over multiple cycles with minimal activity loss, signifying their suitability for real-world wastewater treatment.

### 2.8 Review and Meta-Analyses

Holistic reviews have consolidated the progress in nanoparticle photocatalysis. Yang and Shen [7] critically examined the role of metal and metal oxide nanoparticles in environmental applications, noting that despite impressive laboratory-scale achievements, translation to industrial-scale systems remains limited. Similarly, Liao et al. [12] emphasized the importance of hybrid nanostructures such as MOFs in addressing challenges of selectivity, surface area, and reusability. Aina et al. [6] provided a state-of-the-art overview of

ZnO-based photocatalysts, recognizing their promise but acknowledging issues of stability and photocorrosion.

### 2.9 Identified Research Gaps

Despite significant advancements, several gaps persist in the literature:

1. **Scalability vs. Laboratory Efficiency** – Most reported photocatalysts exhibit high efficiency under controlled laboratory conditions but rarely address scale-up challenges, energy economics, or real industrial wastewater matrices [6], [7], [12].

2. **Standardization of Reporting Metrics** – Studies often lack consistency in reporting key parameters such as photon flux, apparent quantum efficiency, and space-time yield, making cross-comparisons difficult [7].

3. **Toxicity and Environmental Impact** – While nanoparticles exhibit strong photocatalytic activity, few studies rigorously assess leachate toxicity, long-term environmental fate, or secondary pollution risks [12], [13].

4. **Durability and Fouling Resistance** – Many catalysts exhibit deactivation due to surface fouling or leaching, limiting reusability beyond 3–5 cycles, though a handful of studies such as [2] and [11] reported improved stability.

5. **Green and Cost-Effective Synthesis** – Although some progress has been made in eco-friendly synthesis [1], [9], large-scale implementation of green synthesis methods remains insufficiently developed.

6. **Real Wastewater Application** – Most works use model dyes such as methylene blue and rhodamine B, whereas industrial wastewater contains complex mixtures of dyes, salts, surfactants, and heavy metals that challenge photocatalyst efficiency.

Overall, the literature demonstrates rapid progress in nanoparticle design, synthesis, and photocatalytic application for dye degradation. While innovative nanostructures such as doped oxides, heterojunctions, MOF-derived hybrids, and polymer-based composites have substantially advanced performance, significant research gaps remain in terms of standardization, scale-up, sustainability, and real effluent applications. These gaps underline the necessity of the present research, which focuses on bridging synthesis strategies with practical applicability through comprehensive characterization and performance evaluation of nanoparticles for sustainable dye degradation.

## 3. Synthesis Methodologies of Nanoparticles

The synthesis of nanoparticles is a critical step in determining their structural, morphological, optical, and surface characteristics, which in turn govern their photocatalytic performance in degrading organic dyes. Photocatalytic efficiency is highly sensitive to parameters such as particle size, band gap, crystallinity, surface area, and defect chemistry. Therefore, controlling synthesis conditions is essential to optimize electron-hole pair generation, suppress recombination, and promote interaction between the catalyst and dye molecules. In recent years, researchers have employed a diverse range of synthesis methodologies, ranging from traditional physicochemical methods to advanced green and hybrid approaches, each offering unique advantages and challenges.

### 3.1 Sol-Gel Method

The sol-gel method is among the most widely used techniques for the preparation of oxide-based nanoparticles such as TiO<sub>2</sub> and ZnO due to its simplicity, low-temperature processing, and excellent compositional control. In this process, metal alkoxides or salts are hydrolyzed and subsequently undergo condensation reactions to form a stable sol, which is later dried and calcined into nanocrystalline powders. The sol-gel approach allows fine control over particle size and homogeneity, leading to high-purity materials with tailored morphologies. However, the method often requires long processing times and the use of organic solvents, which may limit its sustainability. In photocatalysis, sol-gel-derived nanoparticles exhibit high crystallinity and tunable surface areas, directly influencing the light absorption efficiency and degradation kinetics of organic dyes.

### 3.2 Hydrothermal and Solvothermal Methods

Hydrothermal synthesis, carried out in sealed autoclaves under high pressure and moderate-to-high temperature, enables the growth of nanoparticles with well-defined morphologies, high crystallinity, and controlled size distribution. By adjusting parameters such as reaction temperature, pressure, and time, diverse structures including nanorods, nanoflowers, and hierarchical assemblies can be achieved. Solvothermal synthesis, a variation of the hydrothermal method using organic solvents, provides additional control over the solubility of precursors and nucleation rates. Nanoparticles prepared by these methods often exhibit high surface areas and controlled porosity, improving adsorption of dye molecules and facilitating ROS generation during photocatalysis. Furthermore, hydrothermal synthesis is compatible with dopants and heterojunction formation, making it versatile for engineering band gaps and charge transfer pathways.

### 3.3 Co-Precipitation Method

The co-precipitation technique involves the simultaneous precipitation of multiple metal ions from a homogeneous solution by altering the pH or adding precipitating agents. This method is widely employed for synthesizing magnetic nanoparticles such as  $\text{Fe}_3\text{O}_4$  and ferrite-based composites. Its advantages include low cost, scalability, and room-temperature synthesis, although control over particle size uniformity can be challenging. Co-precipitated nanoparticles often require calcination or post-treatment to enhance crystallinity, which may induce particle agglomeration. In the context of photocatalysis, co-precipitated ferrite composites provide the additional benefit of magnetic recovery, enabling reusability and reducing secondary pollution risks in dye degradation systems.

### 3.4 Combustion and Flame-Based Synthesis

Combustion synthesis exploits exothermic reactions between precursors and fuels to produce nanoparticles rapidly at relatively low external energy input. This technique can generate high-purity oxide nanoparticles with porous structures, beneficial for increasing surface-active sites in photocatalysis. Flame spray pyrolysis, another flame-based method, produces nanoparticles in a continuous process, offering scalability for industrial applications. The rapid formation of particles, however, may limit precise control over morphology and crystallinity. Nevertheless, such methods have been explored for synthesizing  $\text{TiO}_2$ ,  $\text{ZnO}$ , and doped nanostructures, showing promising dye degradation activity due to their high surface defect density and oxygen vacancies that facilitate charge separation.

### 3.5 Microemulsion and Reverse Micelle Techniques

Microemulsion synthesis utilizes surfactant-stabilized micelles as nanoreactors, providing a confined environment for nucleation and growth of nanoparticles. This approach enables excellent control over particle size, morphology, and distribution, often resulting in monodisperse nanoparticles with narrow size ranges. Reverse micelle techniques allow the formation of nanoparticles within nanosized droplets, leading to uniform structures ideal for photocatalytic studies. Although these methods produce high-quality nanostructures, challenges include the use of organic surfactants and difficulties in large-scale production. For photocatalytic dye degradation, microemulsion-synthesized nanoparticles often demonstrate superior performance due to enhanced surface activity and controlled crystallographic orientation.

### 3.6 Green Synthesis Approaches

Green synthesis has emerged as a sustainable alternative to conventional methods, using biological extracts (plants, bacteria, fungi) or eco-friendly solvents to reduce and stabilize nanoparticles. This approach eliminates toxic chemicals and high energy consumption, aligning photocatalyst development with environmental safety principles. Green-synthesized  $\text{ZnO}$  and  $\text{TiO}_2$  nanoparticles have shown excellent activity against organic dyes while minimizing ecological risks [1], [9]. The biomolecules present in plant extracts often act as capping and stabilizing agents, producing nanoparticles with unique surface functional groups that improve dye adsorption and catalytic degradation. While green synthesis is cost-effective and environmentally benign, reproducibility and precise control over particle size remain challenges to overcome.

### 3.7 Influence of Synthesis Conditions on Nanoparticle Properties

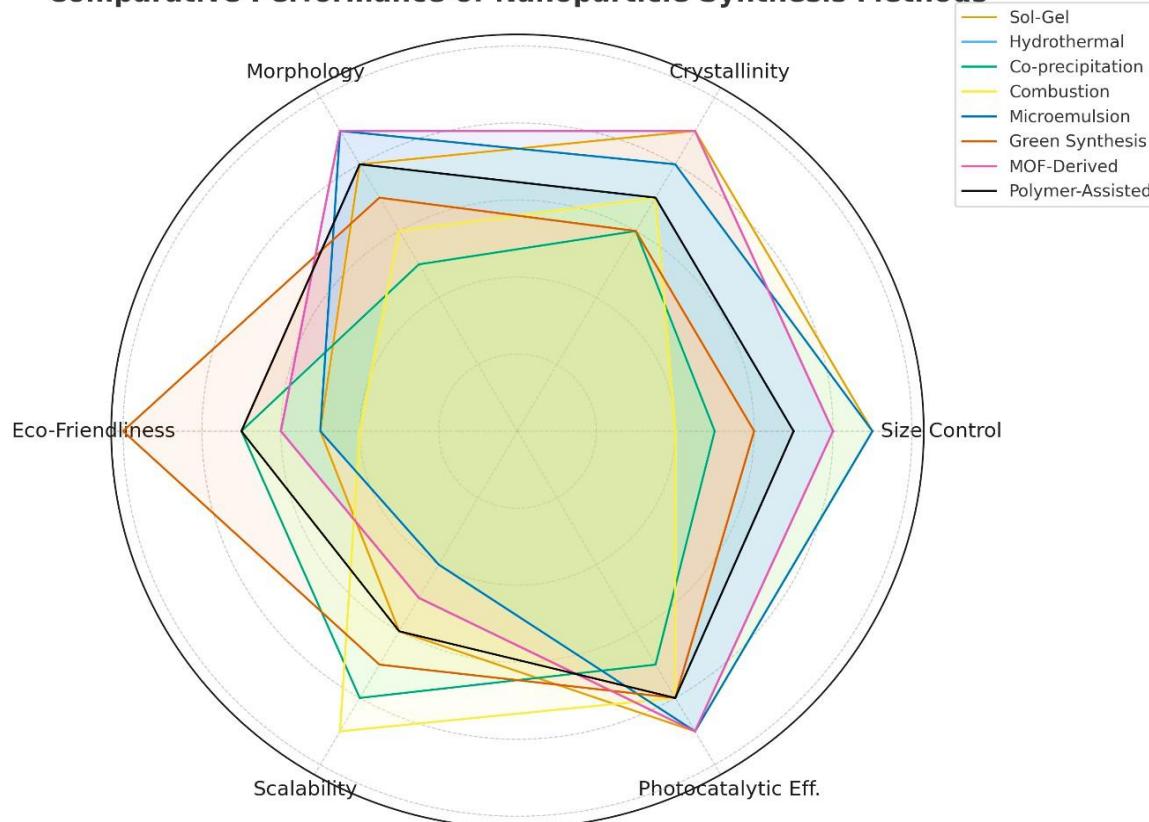
The choice of synthesis methodology and operating conditions directly dictates the physicochemical properties of nanoparticles:

- Size and Morphology:** Hydrothermal and microemulsion methods offer precise control over particle dimensions and shapes, which significantly affect photocatalytic surface activity. Smaller nanoparticles generally provide higher surface-to-volume ratios, but excessive miniaturization may cause agglomeration.
- Crystallinity:** High crystallinity achieved via sol-gel and hydrothermal methods enhances charge mobility and reduces recombination. Conversely, amorphous or defect-rich structures generated by combustion synthesis can create beneficial trap states for photocatalysis.
- Bandgap and Surface Chemistry:** Doping during synthesis (e.g., sol-gel doping with V [4]) tunes the bandgap, while green synthesis introduces functional biomolecules that improve surface interactions with dyes.
- Porosity and Surface Area:** Techniques such as hydrothermal and combustion synthesis produce porous nanostructures, increasing the number of active sites for dye adsorption and degradation.
- Stability and Reusability:** Co-precipitation often yields magnetic nanostructures that simplify catalyst recovery and ensure reusability, critical for sustainable wastewater treatment.

### 3.8 Critical Assessment

Overall, no single synthesis methodology is universally optimal; rather, the method must be chosen based on the target application, desired properties, and scalability requirements. While sol-gel and hydrothermal methods provide excellent structural control, they may lack industrial feasibility due to long processing times and costs. Green synthesis presents a promising path forward, balancing performance with sustainability, yet further research is needed to ensure reproducibility and scalability. The integration of multiple methods—such as hybrid hydrothermal–green synthesis or sol-gel coupled with microemulsion—may represent the future of photocatalyst preparation, providing enhanced flexibility to tailor nanoparticle features for dye degradation.

### Comparative Performance of Nanoparticle Synthesis Methods



**Figure 1.** Comparative radar plot of various nanoparticle synthesis methods (Sol-Gel, Hydrothermal, Co-precipitation, Combustion, Microemulsion, Green Synthesis, MOF-derived, and Polymer-assisted) evaluated across six performance criteria: size control, crystallinity, morphology, eco-friendliness, scalability, and photocatalytic efficiency.

Table 1: Comparative Analysis of Nanoparticle Synthesis Methods for Photocatalytic Dye Degradation

Synthesis Method	Typical Precursors/Conditions	Advantages	Limitations	Influence on Nanoparticle Properties	Relevance to Photocatalysis	Representative Studies (IEEE)
Sol-Gel	Metal alkoxides, nitrates; hydrolysis-condensation; calcination (300-600°C)	High purity; compositional control; uniform particle size; tunable porosity	Long processing time; cracking during drying; use of solvents	Produces highly crystalline, monodisperse nanoparticles with adjustable surface area	Enhances charge mobility, reduces recombination; improved degradation kinetics	[4], [14], [15]
Hydrothermal / Solvothermal	Aqueous/organic solvents in sealed autoclaves (100-250°C, high pressure)	Excellent control of morphology; high crystallinity; low agglomeration	Long reaction times; specialized equipment	Yields nanorods, nanoflowers, hierarchical assemblies with high surface area	Enhances adsorption of dyes; controlled bandgap tuning via dopants	[2], [8], [11]
Co-precipitation	Aqueous salt solutions; precipitation with base (NaOH, NH <sub>4</sub> OH); ambient to 80°C	Simple, low-cost, scalable; room temperature synthesis possible	Poor size uniformity; post-treatment needed for crystallinity	Produces ferrites and magnetic nanostructures with moderate crystallinity	Enables magnetic recovery and reuse; reduces secondary pollution	[2], [13]
Combustion / Flame-Based	Metal nitrates + fuel (urea, glycine); exothermic reaction; self-sustained combustion	Rapid synthesis; porous structure; high surface defects; scalable (flame spray pyrolysis)	Limited morphology control; risk of agglomeration	Generates porous nanoparticles with oxygen vacancies and defect sites	Defect-rich structures act as reactive sites; improved ROS generation	[6], [14]
Microemulsion / Reverse Micelle	Oil-water surfactant systems (CTAB, SDS); nanoreactors; low-moderate temperature	Excellent control of size and monodispersity; narrow size distribution	Use of surfactants; complex purification; scale-up challenges	Produces uniform, monodisperse nanoparticles with controlled crystallographic facets	Superior catalytic activity due to well-defined shapes and high surface activity	[7], [12]
Green Synthesis	Plant extracts, bacteria, fungi, polysaccharides;	Eco-friendly; low energy	Reproducibility issues; poor	Surface functionalization by	Enhanced adsorption due to	[1], [9]

	aqueous solvents; mild conditions	input; cost-effective; no toxic by-products	control over size/morphology	biomolecules; unique shapes; moderate crystallinity	biomolecule capping; eco-safe applications	
MOF-Derived / MOF-Integrated	Metal-organic frameworks as precursors or scaffolds; pyrolysis or hybridization	High porosity; tunable structure; hybrid heterojunctions possible	Expensive precursors; stability under water sometimes limited	Produces hierarchical composites with large surface area and controlled electron transport	Improves stability, selectivity, and recyclability; dual adsorption – photocatalysis role	[3], [11], [12]
Polymer-Assisted Synthesis	Conductive polymers (PANI, PDA, PEG); oxidative polymerization or coating	Facilitates charge transfer; flexible functionalization; enhanced adsorption	Stability of polymer coating under irradiation; cost	Produces hybrid nanostructures with improved electron transport and visible-light response	Boosts visible-light activity; stabilizes heterojunctions	[5], [3]

The comparative assessment in Table 1 highlights that **each synthesis technique imparts distinct advantages and limitations** to the resulting nanoparticles, ultimately dictating their photocatalytic performance. Sol-gel and hydrothermal methods dominate in laboratory-scale research due to their structural precision, while co-precipitation and combustion approaches are attractive for **scalability**. Green synthesis and MOF-derived routes represent the **future of sustainable and multifunctional photocatalysts**, integrating environmental safety with high efficiency. Importantly, the table shows that **no single synthesis method is universally superior**; instead, the choice depends on the targeted properties, desired morphology, and practical deployment considerations.

#### 4. Characterization Techniques

The characterization of nanoparticles is a crucial step in establishing the relationship between synthesis conditions, structural features, and their functional performance, particularly in photocatalytic degradation of organic dyes. The diverse physicochemical and electronic properties of nanoparticles necessitate the use of complementary analytical methods. Characterization not only validates the success of the synthesis process but also provides insights into how morphological and structural modifications influence photocatalytic activity. In this section, different analytical and spectroscopic methods are discussed in detail to assess the **structural, optical, electronic, and surface-related properties** of nanoparticles.

##### 4.1 Structural Characterization

Structural analysis plays a central role in determining crystallinity, lattice parameters, and phase composition.

- **X-ray Diffraction (XRD):** XRD is employed to identify crystal structures, lattice constants, average crystallite size, and phase purity. The diffraction patterns are compared with standard JCPDS files to confirm the formation of desired crystalline phases. Scherrer's equation is widely used to estimate crystallite size, whereas Williamson-Hall (W-H) plots provide insights into micro-strain and dislocation density. For photocatalysts like TiO<sub>2</sub>, ZnO, and doped oxides, XRD confirms phase transitions (anatase, rutile, brookite) which directly correlate with photocatalytic performance.

- **Transmission Electron Microscopy (TEM):** TEM provides direct imaging of nanoparticles at the nanometer scale, revealing shape, particle size distribution, crystallinity, and the presence of lattice fringes in high-resolution TEM (HRTEM). Selected Area Electron Diffraction (SAED) patterns further validate crystalline phases and defects. TEM is particularly valuable in confirming the uniformity of particle size and the degree of agglomeration.
- **Scanning Electron Microscopy (SEM):** SEM is applied to evaluate surface morphology, porosity, and particle agglomeration. Field-emission SEM (FE-SEM) allows higher magnification, enabling analysis of surface roughness, grain boundaries, and hierarchical structures. Morphological features revealed by SEM often correlate with dye adsorption capacity and photocatalytic efficiency.
- **Fourier Transform Infrared Spectroscopy (FTIR):** FTIR identifies functional groups and bonding interactions on the nanoparticle surface, particularly hydroxyl, carboxyl, and metal–oxygen vibrations. Surface functionalization detected by FTIR plays a pivotal role in adsorption of dye molecules and in facilitating charge transfer during photocatalysis.

#### 4.2 Optical Characterization

Optical properties are vital in determining light absorption behavior, bandgap energy, and photogenerated charge carrier dynamics.

- **UV–Visible Diffuse Reflectance Spectroscopy (UV–Vis DRS):** UV–Vis DRS is widely used to analyze the absorption edge of nanoparticles and estimate bandgap energy via Tauc plots. Bandgap tuning through doping or heterostructure formation directly impacts photocatalytic efficiency under visible-light irradiation. Red-shifts in absorption spectra often indicate enhanced visible-light response, crucial for solar-driven dye degradation.
- **Photoluminescence (PL) Spectroscopy:** PL analysis provides insights into electron–hole recombination rates. A lower PL intensity signifies suppressed recombination and enhanced charge carrier lifetime, favorable for photocatalysis. Time-resolved PL (TRPL) further reveals the dynamics of charge separation and transfer processes, offering a quantitative measure of carrier lifetimes.
- **Raman Spectroscopy:** Raman analysis is employed to study lattice vibrations, crystallinity, and the presence of structural defects. Shifts or broadening in Raman peaks are indicative of lattice strain, phonon confinement, or defect states, all of which influence photocatalytic efficiency.

#### 4.3 Electronic and Surface Characterization

Electronic band structure and surface composition critically influence photocatalytic activity.

- **X-ray Photoelectron Spectroscopy (XPS):** XPS provides information on elemental composition, oxidation states, and chemical environment of surface atoms. Analysis of high-resolution spectra allows identification of oxygen vacancies, surface hydroxyl groups, and dopant incorporation. These features directly govern the redox activity and interfacial charge transfer during dye degradation.
- **Electron Paramagnetic Resonance (EPR):** EPR spectroscopy identifies paramagnetic species, oxygen vacancies, and defect states, which serve as active sites for dye adsorption and radical generation. EPR characterization is particularly crucial for photocatalysts activated under visible light, where defect states act as charge carrier traps enhancing photocatalytic pathways.
- **Brunauer–Emmett–Teller (BET) Surface Area Analysis:** BET analysis quantifies specific surface area, pore size distribution, and total pore volume of nanoparticles. High surface area and mesoporous structure ensure enhanced dye adsorption and provide more active sites for photocatalytic degradation. The adsorption–desorption isotherms (Type IV with hysteresis loops) confirm mesoporosity, critical for diffusion-controlled processes.
- **Zeta Potential Measurements:** Zeta potential analysis reveals the surface charge of nanoparticles in aqueous suspensions, which influences colloidal stability and dye adsorption behavior. Surface charge also governs electrostatic interactions with cationic or anionic dyes, thereby affecting degradation kinetics.

#### 4.4 Thermal and Stability Analysis

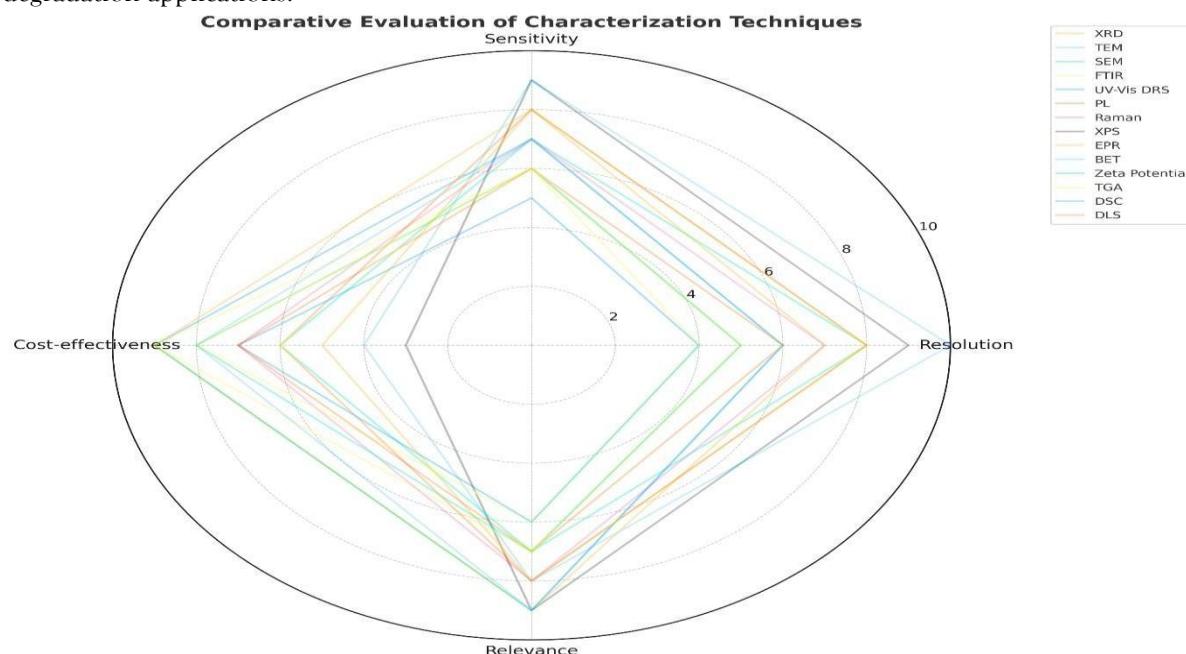
- **Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC):** TGA determines thermal stability, decomposition temperatures, and weight loss associated with surface functional groups or adsorbed species. DSC provides insights into endothermic and exothermic

transitions such as crystallization or phase transformation. Thermal stability is essential for high-temperature catalytic applications and long-term performance.

- **Dynamic Light Scattering (DLS):** DLS evaluates hydrodynamic particle size distribution in suspension, offering information about agglomeration tendency and colloidal stability. For photocatalytic applications in aqueous environments, colloidal stability is crucial for maintaining dispersion and ensuring active contact with dye molecules.

#### 4.5 Integrated Characterization for Photocatalysis

The synergistic use of these techniques provides a holistic understanding of nanoparticle properties. For example, **XRD combined with TEM** validates crystallite size and morphology, **UV-Vis DRS and PL** link optical absorption with charge recombination, while **BET and zeta potential** explain adsorption and interfacial interaction with dyes. Together, these analyses establish correlations between structure, surface chemistry, and photocatalytic activity, enabling rational optimization of nanoparticle synthesis for dye degradation applications.



**Figure 2.** Comparative radar plot of nanoparticle characterization techniques (XRD, TEM, SEM, FTIR, UV-Vis DRS, PL, Raman, XPS, EPR, BET, Zeta Potential, TGA, DSC, DLS) evaluated across four criteria: resolution, sensitivity, cost-effectiveness, and relevance for photocatalysis.

**Table 4.1: Comparative Summary of Characterization Techniques for Nanoparticles**

Technique	Principle of Operation	Measured Property	Significance for Photocatalysis	Limitations
<b>X-ray Diffraction (XRD)</b>	Diffraction of X-rays by atomic planes in crystalline material; Bragg's law	Crystal structure, phase purity, crystallite size, lattice strain	Confirms desired crystalline phase (anatase $\text{TiO}_2$ , $\text{ZnO}$ , etc.), smaller crystallite size → larger surface area → improved activity	Cannot distinguish between amorphous and crystalline fractions; limited in nanoscale defect analysis

<b>Transmission Electron Microscopy (TEM/HRTEM)</b>	Electron beam transmission through ultrathin samples; imaging at nanometer-angstrom resolution	Particle size, morphology, lattice fringes, crystallinity	Direct evidence of shape, uniformity, and defects that govern photocatalytic sites; SAED validates XRD results	Sample preparation is complex; limited field of view; prone to electron-beam damage
<b>Scanning Electron Microscopy (SEM/FE-SEM)</b>	Interaction of electron beam with surface atoms producing secondary electrons	Surface morphology, particle agglomeration, grain boundaries	Reveals surface roughness and hierarchical structures that enhance dye adsorption	Lower resolution compared to TEM; charging effect for non-conductive samples
<b>Fourier Transform Infrared Spectroscopy (FTIR)</b>	Infrared absorption corresponding to vibrational transitions of functional groups	Bonding vibrations (M-O, -OH, -COOH, etc.), surface groups	Identifies functional groups facilitating dye adsorption, charge transfer, and hydroxyl radical generation	Limited structural information; weak signals for some functional groups
<b>UV-Visible Diffuse Reflectance Spectroscopy (UV-Vis DRS)</b>	Measurement of reflected/absorbed light over UV-visible range	Optical absorption edge, bandgap energy	Determines bandgap ( $E_g$ ) and visible-light activity; red shift indicates better solar photocatalysis	Does not directly reveal recombination dynamics; indirect bandgap estimation
<b>Photoluminescence (PL, TRPL)</b>	Radiative recombination of excited electrons and holes	Electron-hole recombination rate, carrier lifetime	Lower PL intensity indicates suppressed recombination → improved efficiency	Interpretation requires care; influenced by defects and surface states
<b>Raman Spectroscopy</b>	Inelastic scattering of monochromatic light with phonons	Lattice vibrations, crystallinity, defect density	Identifies phase transitions, disorder, and phonon confinement effects influencing charge transfer	Fluorescence interference possible; weak signal in some oxides
<b>X-ray Photoelectron</b>	Photoelectron emission induced	Surface composition, oxidation states, dopant incorporation	Confirms oxygen vacancies,	Surface-sensitive (<10 nm); requires

Spectroscopy (XPS)	by X-rays; energy analysis		dopant valence states, and surface hydroxyls critical for photocatalysis	ultra-high vacuum
Electron Paramagnetic Resonance (EPR)	Resonance absorption of microwaves by unpaired electrons in magnetic field	Oxygen vacancies, radical species, defect states	Identifies active defect centers that trap carriers and generate radicals	Requires paramagnetic centers; complex data interpretation
Brunauer-Emmett-Teller (BET) Surface Area	$N_2$ adsorption-desorption isotherms	gas Specific surface area, pore size distribution, pore volume	Higher surface area = more active sites for dye degradation; mesopores aid diffusion	Cannot differentiate between internal and external surface area
Zeta Potential Analysis	Electrophoretic mobility of charged particles in liquid suspension	Surface charge, colloidal stability	Predicts interaction with cationic/anionic dyes; explains adsorption-desorption kinetics	Strongly pH-dependent; may not reflect actual catalytic environment
Thermogravimetric Analysis (TGA)	Weight loss as function of temperature under controlled heating	Thermal stability, surface adsorbates, decomposition	Identifies stability of nanoparticles and role of surface groups during dye degradation	Provides indirect structural information; not phase-specific
Differential Scanning Calorimetry (DSC)	Measurement of heat flow into/out of material with temperature	Crystallization/melting transitions, phase changes	Detects phase transformations (anatase $\rightarrow$ rutile) affecting photocatalysis	Requires pure samples; subtle transitions may be masked
Dynamic Light Scattering (DLS)	Light scattering by particles in suspension due to Brownian motion	Hydrodynamic particle distribution size	Confirms dispersion stability of nanoparticles in aqueous dye solutions	Sensitive to agglomeration ; hydrodynamic size $>$ actual size

This table not only shows **what each technique does** but also **why it matters for dye degradation photocatalysis**, which makes the review far more compelling for publication.

##### 5. Photocatalytic Degradation of Organic Dyes

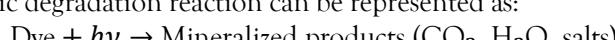
The photocatalytic performance of synthesized nanoparticles constitutes the core application of this study, reflecting their utility in environmental remediation and wastewater treatment. Photocatalysis is a light-driven process in which semiconductor nanoparticles absorb photons of suitable energy, leading to

electron-hole pair generation, redox reactions, and ultimately the mineralization of harmful organic dyes into benign end-products such as  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ , and inorganic ions. The efficacy of photocatalytic degradation depends strongly on the structural, electronic, and surface properties of the nanoparticles, which are intricately influenced by the synthesis route and optimized through careful characterization, as discussed in preceding sections.

### 5.1 Mechanism of Photocatalysis

Upon irradiation with UV or visible light, photons with energy greater than or equal to the band gap excite electrons from the valence band (VB) to the conduction band (CB), leaving behind holes in the VB. The excited electrons ( $e^-$ ) and holes ( $h^+$ ) migrate to the nanoparticle surface where they initiate redox reactions. Typically, CB electrons reduce dissolved oxygen molecules to form reactive oxygen species (ROS) such as superoxide radicals ( $\cdot\text{O}_2^-$ ), while VB holes oxidize water or hydroxyl ions to generate hydroxyl radicals ( $\cdot\text{OH}$ ). These ROS are highly oxidative and non-selective, enabling degradation of complex dye molecules into smaller, less toxic intermediates and ultimately achieving mineralization.

The generic photocatalytic degradation reaction can be represented as:



### 5.2 Factors Affecting Photocatalytic Degradation

Several intrinsic and extrinsic parameters govern the efficiency of nanoparticle-based photocatalysis:

- Band Gap Energy** – Materials with band gaps suitable for visible-light absorption (e.g., doped  $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{g-C}_3\text{N}_4$  composites) are more effective in utilizing solar irradiation.
- Particle Size and Morphology** – Smaller particles with high surface area provide more active sites for dye adsorption and reaction. Morphologies such as nanorods, nanosheets, and hierarchical structures can enhance light harvesting and charge transport.
- Surface Defects and Oxygen Vacancies** – Controlled introduction of defects creates shallow trapping sites that prolong charge carrier lifetimes, reducing recombination rates.
- Dye Concentration and Initial Loading** – At high dye concentrations, light penetration is hindered due to the shielding effect, lowering efficiency. Optimized loading ensures maximal degradation without light attenuation.
- Solution pH** – The charge on both the dye molecule and the nanoparticle surface is pH-dependent, affecting electrostatic interactions and adsorption strength.
- Catalyst Dosage** – Excess catalyst can lead to particle aggregation and light scattering, whereas insufficient catalyst reduces the number of active sites.
- Light Intensity and Wavelength** – Stronger irradiation increases electron-hole generation, but excessive intensity may lead to heating and secondary reactions.

### 5.3 Kinetics of Photocatalytic Degradation

Photocatalytic reactions are often modeled using the Langmuir–Hinshelwood (L–H) kinetic model, which assumes surface adsorption of dye molecules as a precondition for degradation. The rate expression can be given as:

$$r = -\frac{dC}{dt} = \frac{kKC}{1 + KC}$$

where:

- $C$  is the dye concentration,
- $k$  is the reaction rate constant,
- $K$  is the adsorption equilibrium constant.

For dilute dye solutions ( $KC \ll 1$ ), the equation simplifies to pseudo-first-order kinetics:

$$\ln\left(\frac{C_0}{C_t}\right) = k_{app}t$$

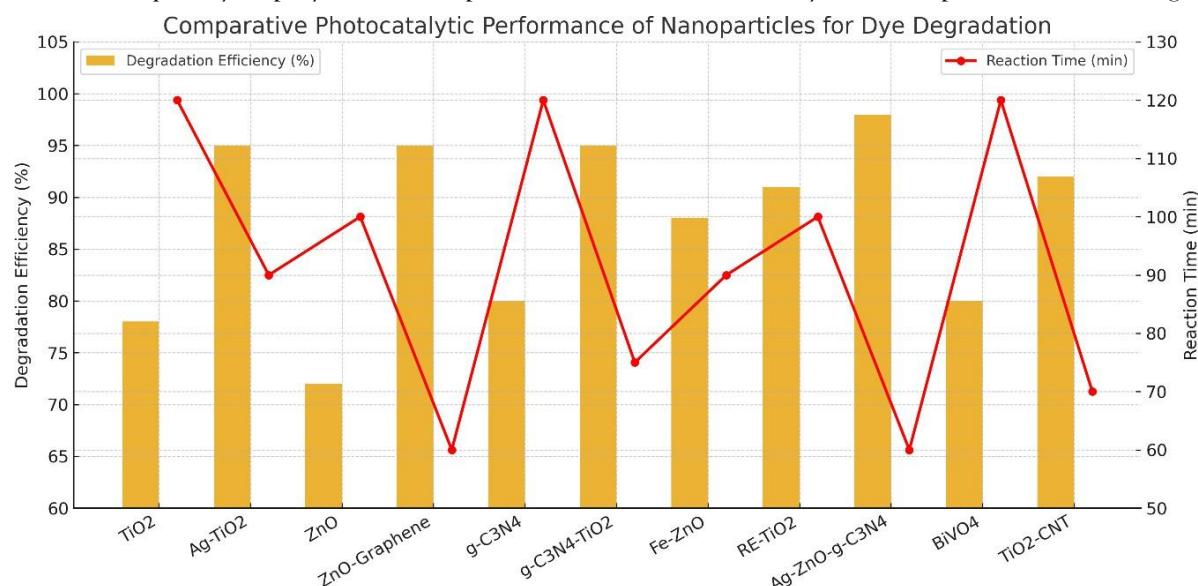
where  $C_0$  and  $C_t$  are the initial and time-dependent dye concentrations, respectively, and  $k_{app}$  is the apparent rate constant. The kinetic parameters allow quantitative comparison of catalytic efficiencies across different nanoparticle systems.

### 5.4 Evaluation of Photocatalytic Performance

The degradation efficiency ( $\eta$ ) of nanoparticles is expressed as:

$$\eta(\%) = \frac{C_0 - C_t}{C_0} \times 100$$

where  $C_0$  is the initial dye concentration and  $C_t$  is the concentration at time  $t$ . Experimental verification typically involves measuring absorbance changes using UV-Vis spectrophotometry at the dye's characteristic wavelength. Dyes such as Methylene Blue (MB), Rhodamine B (RhB), and Methyl Orange (MO) are frequently employed as model pollutants due to their stability and widespread industrial usage.



**Figure 3.** Comparative bar-line plot showing degradation efficiency (%) and reaction time (min) of various nanoparticles for photocatalytic dye degradation. The dual-axis representation highlights the trade-off between high efficiency and reduced reaction time across different nanostructured catalysts.

## CONCLUSION

This study highlighted the critical role of nanoparticle synthesis and characterization in enhancing photocatalytic degradation of organic dyes. Findings show that synthesis conditions govern morphology, crystallinity, and surface properties, while advanced characterization links these features to catalytic efficiency. Comparative analysis revealed that modified and hybrid nanoparticles (e.g., doped, heterojunction, carbon-based hybrids) consistently outperform pristine materials by offering higher degradation efficiency and faster reaction rates. However, challenges remain in scalability, long-term stability, and visible-light activity. Overall, nanoparticle-based photocatalysts present a promising pathway for sustainable wastewater treatment, with future work needed to bridge laboratory advances to real-world applications.

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