IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429
Volume 5, Issue 5, December 2025
Impact Factor: 7.67

Review on Synthesis Methods on the Photocatalytic Performance of Zinc Oxide and Iron Oxide Nanoparticles

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Abstract: *Nanotechnology has gained significant attention in environmental remediation due to the exceptional photocatalytic properties of metal oxide nanoparticles. Among these, zinc oxide and iron oxide (Fe_2O_3/Fe_3O_4) nanoparticles have demonstrated remarkable potential in the degradation of organic pollutants under light irradiation. The photocatalytic efficiency of these nanoparticles is highly dependent on their synthesis method, which influences particle size, surface area, crystallinity, and morphology. This review summarizes recent developments in various synthesis techniques for ZnO and Fe oxide nanoparticles and their impact on photocatalytic activity.*

Keywords: Zinc oxide nanoparticles, Iron oxide nanoparticles, Photocatalysis

I. INTRODUCTION

Photocatalysis is a promising technique for the degradation of environmental pollutants, relying on semiconductor materials capable of generating electron–hole pairs under light irradiation. ZnO and Fe oxide nanoparticles are widely studied due to their stability, low cost, and strong photocatalytic activity (Zhang et al., 2020; Sharma et al., 2019). The choice of synthesis method plays a crucial role in determining structural properties, surface area, and defect density, which directly affect photocatalytic performance (Kumar et al., 2021).

Nanotechnology has revolutionized environmental remediation and energy conversion technologies due to the unique physicochemical properties of nanoparticles. Among various nanomaterials, metal oxide nanoparticles, particularly zinc oxide and iron oxide (Fe_2O_3/Fe_3O_4), have emerged as promising photocatalysts for the degradation of organic pollutants in water and air. Photocatalysis is a process in which semiconductor materials absorb light energy to generate electron–hole pairs, which subsequently initiate redox reactions to break down harmful contaminants. The efficiency of photocatalytic reactions is highly dependent on the structural, morphological, and optical properties of the nanoparticles, which are, in turn, influenced by the synthesis methods employed (Zhang, Li, & Sun, 2020).

ZnO nanoparticles are widely studied due to their wide bandgap (~3.37 eV), high exciton binding energy, chemical stability, and biocompatibility, making them effective under ultraviolet light irradiation for degrading dyes, pharmaceuticals, and other organic compounds (Li, Zhang, & Zhou, 2017). Iron oxide nanoparticles, including hematite (Fe_2O_3) and magnetite (Fe_3O_4), offer a narrower bandgap (~2.0–2.2 eV) and visible light activity, enabling their application in solar-driven photocatalysis. Additionally, the magnetic properties of Fe_3O_4 facilitate easy recovery and reuse of photocatalysts, which is advantageous for sustainable environmental applications (Gupta & Sharma, 2020).

The synthesis method plays a pivotal role in determining particle size, crystallinity, surface area, porosity, and defect density, all of which critically affect photocatalytic performance. Various chemical, physical, and biological routes have been employed to synthesize ZnO and Fe oxide nanoparticles with controlled morphology and enhanced functionality. Chemical precipitation and sol–gel methods offer simplicity, low cost, and the ability to control particle size and homogeneity (Ramesh, Kumar, & Thomas, 2018). Hydrothermal and solvothermal techniques allow precise tuning of shape, size, and crystallinity under controlled temperature and pressure conditions, which significantly enhances surface-to-volume ratios and photocatalytic activity (Zhou, Wu, & Yang, 2018; Singh, Kumar, & Choudhary, 2020).

Green synthesis using plant extracts, microbes, or biopolymers has emerged as an eco-friendly approach that minimizes toxic reagents while producing nanoparticles with high stability and photocatalytic efficiency (Khan, Ahmed, & Fatima, 2021). Furthermore, advanced techniques such as microwave-assisted and sonochemical synthesis have been explored to accelerate nucleation, achieve uniform particle distribution, and improve light absorption, thereby optimizing photocatalytic activity (Chen, Zhang, & Liu, 2019).

Understanding the relationship between synthesis methods, nanoparticle properties, and photocatalytic efficiency is crucial for designing high-performance materials for environmental remediation. The ongoing research focuses not only on optimizing single-metal oxide nanoparticles but also on developing heterostructures and composite systems, such as ZnO/Fe_2O_3, which enhance charge separation, reduce electron–hole recombination, and improve degradation efficiency under both UV and visible light (Patil, Deshmukh, & Jadhav, 2019). This review aims to summarize the recent advances in synthesis strategies for ZnO and Fe oxide nanoparticles and analyze their impact on photocatalytic performance, highlighting key factors that govern activity and providing insights for future research in sustainable photocatalysis.

SYNTHESIS METHODS

The synthesis of zinc oxide and iron oxide (Fe_2O_3/Fe_3O_4) nanoparticles is a critical factor influencing their structural, optical, and photocatalytic properties, as these characteristics are directly dependent on particle size, morphology, crystallinity, and surface defects. Among the most widely employed techniques, chemical precipitation stands out for its simplicity, cost-effectiveness, and scalability. In this method, ZnO nanoparticles are typically synthesized by reacting zinc salts such as zinc acetate or zinc nitrate with a base like sodium hydroxide, leading to the formation of ZnO nuclei, which grow into nanoparticles under controlled conditions.

Precipitation parameters such as pH, temperature, and reactant concentration significantly influence the size and shape of the nanoparticles, and smaller, well-dispersed particles exhibit higher surface areas, promoting enhanced photocatalytic activity (Ramesh et al., 2018). Similarly, iron oxide nanoparticles, particularly Fe_3O_4, are synthesized via co-precipitation, where ferric and ferrous salts react in an alkaline medium. This method yields magnetic nanoparticles with uniform size distribution, which have shown considerable photocatalytic efficiency in the degradation of organic pollutants due to their high surface reactivity and ability to generate reactive oxygen species (Gupta & Sharma, 2020).

The sol–gel method is another versatile approach for nanoparticle synthesis, offering precise control over composition and homogeneity at relatively low temperatures. In this process, metal alkoxides or metal salts are hydrolyzed to form a colloidal sol, which gradually transforms into a gel and, upon calcination, yields crystalline nanoparticles. ZnO nanoparticles synthesized via sol–gel typically exhibit high crystallinity, controlled particle size, and enhanced surface activity, resulting in superior photocatalytic performance in the degradation of dyes and phenolic compounds (Li et al., 2017). For iron oxides, sol–gel synthesis produces porous Fe_2O_3 nanoparticles with tunable size and morphology, enabling improved light absorption and charge separation, which are critical for efficient photocatalysis under visible light irradiation (Patil et al., 2019).

Hydrothermal and solvothermal methods have gained prominence due to their ability to produce nanoparticles with well-defined shapes and high crystallinity. These techniques involve the reaction of precursors in a sealed, high-pressure autoclave, which facilitates controlled nucleation and growth of nanostructures. ZnO nanorods, nanowires, and hierarchical structures synthesized hydrothermally possess high aspect ratios and surface areas, significantly improving their photocatalytic activity under UV irradiation (Zhou et al., 2018). Similarly, Fe_2O_3 nanoflakes or nanospheres prepared hydrothermally exhibit enhanced electron–hole separation and light absorption, leading to effective degradation of organic contaminants (Singh et al., 2020).

In recent years, green synthesis approaches using plant extracts, microbial metabolites, or biopolymers have emerged as eco-friendly alternatives. These methods reduce the need for hazardous chemicals and offer mild reaction conditions. ZnO and Fe oxide nanoparticles synthesized via green routes not only show comparable photocatalytic activity to chemically synthesized nanoparticles but also minimize environmental impact, making them suitable for sustainable applications in wastewater treatment and pollutant degradation (Khan et al., 2021).

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Other techniques such as microwave-assisted synthesis, sonochemical methods, and chemical vapor deposition provide rapid nucleation, uniform particle distribution, and high purity, further enhancing photocatalytic performance (Chen et al., 2019). Overall, the choice of synthesis method strongly determines nanoparticle morphology, crystallinity, and surface characteristics, which in turn govern their photocatalytic efficiency. Understanding these correlations is essential for optimizing ZnO and Fe oxide nanoparticles for environmental remediation and sustainable photocatalytic applications.

CHEMICAL PRECIPITATION

Chemical precipitation is a simple and cost-effective method for producing ZnO and Fe oxide nanoparticles. Controlled precipitation allows the tuning of particle size and morphology. For instance, ZnO nanoparticles synthesized via precipitation with NaOH showed enhanced photocatalytic degradation of methylene blue due to increased surface area (Ramesh et al., 2018). Similarly, Fe_3O_4 nanoparticles prepared by co-precipitation demonstrated high magnetic properties and effective photocatalytic activity (Gupta & Sharma, 2020).

Chemical Precipitation is one of the most widely employed and straightforward methods for synthesizing metal oxide nanoparticles, including zinc oxide (ZnO) and iron oxide (Fe_2O_3/Fe_3O_4), due to its simplicity, cost-effectiveness, and ability to control particle size and morphology. In this method, metal salts such as zinc acetate, zinc nitrate, ferric chloride, or ferrous sulfate are dissolved in a suitable solvent, and a precipitating agent, typically sodium hydroxide or ammonium hydroxide, is added to induce nucleation and particle formation (Ramesh et al., 2018; Gupta & Sharma, 2020).

The reaction parameters, including pH, temperature, reactant concentration, and stirring rate, significantly influence the size, shape, and crystallinity of the resulting nanoparticles. For instance, ZnO nanoparticles synthesized via chemical precipitation under alkaline conditions often exhibit spherical or rod-like morphologies with high surface areas, which enhances their interaction with pollutants and improves photocatalytic efficiency under UV light (Ramesh et al., 2018).

Similarly, Fe_3O_4 nanoparticles produced through co-precipitation demonstrate superparamagnetic properties, allowing easy recovery after photocatalytic applications while maintaining high degradation efficiency of organic dyes under visible light (Gupta & Sharma, 2020). The simplicity of this method also allows for the incorporation of dopants or surface modifiers to tailor bandgap energy and reduce electron–hole recombination, thereby enhancing photocatalytic performance.

However, challenges such as particle agglomeration, broad size distribution, and limited control over morphology may arise, requiring post-synthesis treatments such as calcination or surfactant-assisted growth to improve stability and surface characteristics. Despite these limitations, chemical precipitation remains a preferred approach for large-scale and eco-friendly production of ZnO and Fe oxide nanoparticles due to its scalability, low energy requirements, and reproducibility, making it a vital technique in environmental remediation and photocatalytic applications.

SOL–GEL METHOD

The sol–gel process offers precise control over composition and homogeneity at low temperatures. ZnO nanoparticles synthesized by sol–gel exhibited high crystallinity and smaller particle size, which improved photocatalytic efficiency (Li et al., 2017). Fe_2O_3 nanoparticles produced by sol–gel showed enhanced light absorption and faster degradation of organic dyes (Patil et al., 2019).

The sol–gel method has emerged as one of the most versatile and widely used techniques for synthesizing metal oxide nanoparticles, including zinc oxide (ZnO) and iron oxide (Fe_2O_3/Fe_3O_4), due to its ability to control particle size, morphology, and crystallinity at relatively low temperatures. This method involves the transition of a system from a colloidal "sol" into a solid "gel" phase, typically through hydrolysis and polycondensation of metal alkoxides or metal salts in the presence of a solvent (Li et al., 2017). For ZnO nanoparticles, the sol–gel process allows precise manipulation of structural parameters, which is critical for enhancing photocatalytic activity.

By adjusting parameters such as pH, reaction temperature, precursor concentration, and aging time, researchers have successfully produced ZnO nanoparticles with sizes ranging from 10 to 50 nm and morphologies such as nanospheres, nanorods, and nanoflowers (Ramesh et al., 2018). The high degree of crystallinity and uniform particle distribution achieved through sol–gel synthesis contributes significantly to efficient electron–hole pair generation and reduces the rate of recombination, thus improving photocatalytic degradation of organic pollutants such as methylene blue, rhodamine B, and phenol under UV irradiation (Zhang et al., 2020).

Similarly, iron oxide nanoparticles synthesized via the sol–gel method, particularly hematite (α-Fe_2O_3) and magnetite (Fe_3O_4), exhibit enhanced photocatalytic performance under visible light due to their narrower bandgap energies (2.0–2.2 eV), which enable the absorption of a broader spectrum of sunlight (Patil et al., 2019). The sol–gel approach allows the formation of porous structures and high surface area nanoparticles, which increase the number of active sites available for photocatalytic reactions. Furthermore, the method facilitates doping with metals or coupling with other semiconductors to form heterojunctions, such as ZnO/Fe_2O_3 composites, which further improve photocatalytic efficiency by promoting charge separation and reducing electron–hole recombination (Li et al., 2017; Chen et al., 2019).

One of the advantages of the sol–gel method is its adaptability for large-scale production while maintaining particle uniformity and reproducibility. Moreover, it provides a simple route for surface functionalization and modification, which can enhance stability, dispersibility, and reusability of nanoparticles in photocatalytic applications. For instance, surface-modified ZnO nanoparticles synthesized by sol–gel exhibited higher photocatalytic degradation rates compared to bare nanoparticles due to improved adsorption of pollutants on the nanoparticle surface (Ramesh et al., 2018). In addition, the low-temperature processing characteristic of the sol–gel method prevents aggregation and preserves the nanoscale features that are critical for photocatalysis.

The sol–gel method remains a highly effective and controllable synthesis route for producing ZnO and iron oxide nanoparticles with superior photocatalytic performance. By enabling fine-tuning of particle size, shape, surface area, and crystallinity, sol–gel-synthesized nanoparticles demonstrate enhanced degradation of organic pollutants, making them highly suitable for environmental remediation applications. Future research is likely to focus on optimizing sol–gel parameters, exploring novel dopants, and developing hybrid systems to further boost the photocatalytic efficiency of these nanoparticles.

HYDROTHERMAL/SOLVOTHERMAL METHODS

Hydrothermal synthesis enables the production of nanoparticles with controlled shape, size, and crystallinity. ZnO nanorods and Fe_2O_3 nanoflakes synthesized hydrothermally exhibited high surface-to-volume ratios, increasing photocatalytic performance under UV and visible light (Zhou et al., 2018; Singh et al., 2020).

GREEN SYNTHESIS

Green synthesis using plant extracts or biopolymers has gained attention as an eco-friendly approach. ZnO and Fe oxide nanoparticles synthesized using plant-mediated methods demonstrated good photocatalytic activity while minimizing toxic by-products (Khan et al., 2021).

OTHER METHODS

Other techniques, such as microwave-assisted synthesis, sonochemical methods, and chemical vapor deposition, have also been explored. These methods provide rapid nucleation, uniform particle distribution, and high purity, contributing to enhanced photocatalytic degradation rates (Chen et al., 2019). In addition to conventional synthesis approaches such as chemical precipitation, sol–gel, hydrothermal, and green synthesis, several alternative techniques have emerged to improve the photocatalytic performance of ZnO and iron oxide nanoparticles.

Microwave-assisted synthesis has gained significant attention due to its rapid heating, uniform nucleation, and ability to produce nanoparticles with controlled size and morphology. The technique facilitates high crystallinity and minimizes agglomeration, which directly enhances the photocatalytic degradation of organic dyes and pollutants (Chen, Zhang, & Liu, 2019). Microwave irradiation reduces reaction time from several hours, as seen in conventional hydrothermal methods, to mere minutes, making it an efficient and scalable process for industrial applications.

Similarly, sonochemical synthesis, which uses ultrasonic waves to induce cavitation in the reaction medium, has been widely applied for ZnO and Fe oxide nanoparticle preparation. The intense local temperature and pressure generated during cavitation promote rapid nucleation and the formation of smaller, uniformly dispersed nanoparticles. ZnO nanoparticles prepared via sonochemical routes exhibit enhanced photocatalytic activity due to increased surface area and defect sites that facilitate electron–hole separation (Kumar et al., 2021). Iron oxide nanoparticles synthesized using sonochemistry have also demonstrated superior photocatalytic degradation of phenolic compounds and dyes under visible light, highlighting the method's versatility.

Chemical vapor deposition is another alternative method that offers precise control over nanoparticle composition, size, and surface properties. CVD allows the deposition of ZnO and Fe oxide nanoparticles onto various substrates, enabling the fabrication of thin films and heterostructures that enhance photocatalytic efficiency. The technique produces highly crystalline nanoparticles with minimal impurities, which are essential for stable and reproducible photocatalytic performance (Patil et al., 2019). CVD-synthesized nanoparticles also allow the engineering of specific morphologies, such as nanowires or nanoflowers, which significantly increase the surface-to-volume ratio and improve the adsorption of target pollutants.

Electrochemical deposition and electrodeposition methods have also been explored for producing ZnO and iron oxide nanoparticles. These methods provide a straightforward and cost-effective route to synthesize nanoparticles directly on conductive substrates, offering excellent adhesion and uniformity. ZnO nanoparticles synthesized via electrodeposition show enhanced photocatalytic activity due to their tunable thickness and porosity, which allows for effective light absorption and charge transfer (Li et al., 2017). Similarly, iron oxide thin films prepared electrochemically exhibit controlled phase composition, which is critical for maximizing visible-light-driven photocatalysis.

Lastly, hybrid methods that combine two or more synthesis techniques have shown considerable promise in enhancing photocatalytic efficiency. For example, combining sol–gel and microwave-assisted methods or hydrothermal and green synthesis approaches allows for precise control over particle size, morphology, and defect density, which are crucial factors in improving electron–hole separation and light absorption (Zhang, Li, & Sun, 2020). These advanced synthesis strategies not only enhance photocatalytic performance but also provide environmentally friendly and scalable alternatives for large-scale pollutant remediation applications.

Overall, these other methods, including microwave-assisted, sonochemical, CVD, electrochemical, and hybrid approaches, demonstrate significant potential for producing ZnO and iron oxide nanoparticles with superior structural and photocatalytic properties. By enabling precise control over size, morphology, and surface defects, these techniques contribute to the development of highly efficient photocatalysts for environmental and industrial applications.

PHOTOCATALYTIC PERFORMANCE

The photocatalytic activity of nanoparticles is influenced by several factors, including particle size, morphology, crystallinity, surface defects, and bandgap energy. ZnO nanoparticles generally show higher activity under UV light due to their wide bandgap (~3.37 eV), whereas Fe_2O_3 nanoparticles are more effective under visible light (~2.2 eV) (Ramesh et al., 2018; Gupta & Sharma, 2020). Combining ZnO and Fe oxide nanoparticles into heterostructures further enhances photocatalytic efficiency by improving charge separation and reducing electron–hole recombination.

Table 1: Synthesis Methods and Photocatalytic Performance of ZnO and Fe Oxide Nanoparticles

Nanoparticle	Synthesis Method	Particle Size / Morphology	Photocatalytic Activity	Reference
ZnO	Chemical precipitation	20–50 nm, spherical	High degradation of dyes under UV	Ramesh et al., 2018
ZnO	Sol–gel	10–30 nm, high crystallinity	Enhanced MB degradation under UV	Li et al., 2017
ZnO	Hydrothermal	Nanorods, high aspect ratio	Rapid degradation of phenol	Zhou et al., 2018
ZnO	Green synthesis	15–40 nm, flower-like	Eco-friendly, moderate activity	Khan et al., 2021
Fe_3O_4	Co-precipitation	10–50 nm, magnetic	High photocatalytic efficiency	Gupta & Sharma,

				2020
Fe_2O_3	Sol–gel	20–60 nm, porous flakes	Visible light degradation of dyes	Patil et al., 2019
Fe_2O_3	Hydrothermal	Nanoflakes	Enhanced phenol degradation	Singh et al., 2020
ZnO/Fe_2O_3	Microwave-assisted	10–50 nm, composite	Superior charge separation	Chen et al., 2019

II. CONCLUSION

The synthesis method profoundly impacts the structural, optical, and photocatalytic properties of ZnO and Fe oxide nanoparticles. Chemical precipitation and sol–gel methods are simple and widely used, while hydrothermal and green synthesis methods offer controlled morphology and eco-friendly production. Understanding the relationship between synthesis parameters and photocatalytic performance is critical for designing efficient photocatalysts for environmental remediation applications. Future research should focus on developing hybrid systems and optimizing synthesis conditions for large-scale applications.

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