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REVIEW

Biomass as a Green Source of Dopants: A Review on In-Situ Synthesis of P-N Co-Doped ZnO for Photocatalytic Dye Degradation

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ABSTRACT

Synthetic dyes, particularly azo dyes, pose significant environmental and health risks due to their persistence, toxicity, and potential carcinogenicity. Zinc oxide (ZnO) is a promising photocatalyst for wastewater remediation, but its wide bandgap and rapid charge recombination limit its practical efficacy. Furthermore, conventional doping methods often rely on hazardous chemical precursors, undermining the sustainability of the overall approach. This review introduces a novel and sustainable paradigm: the utilization of biomass-derived precursors as green reagents for the in-situ synthesis and simultaneous phosphorus-nitrogen (P-N) co-doping of ZnO nanoparticles. We critically analyze how the intrinsic biochemical composition of biomass, rich in P, N, and other heteroatoms, facilitates this one-pot, eco-friendly functionalization. This

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ARTICLE INFO

Received: 8 September 2025 | Revised: 2 December 2025 | Accepted: 16 December 2025 | Published Online: 5 January 2026
DOI: https://doi.org/10.30564/jees.v8i1.11970

CITATION

Matlaila, M.M., Khumalo, N.L., Mohomane, S.M., et al., 2026. Biomass as a Green Source of Dopants: A Review on In-Situ Synthesis of P-N Co-Doped ZnO for Photocatalytic Dye Degradation. Journal of Environmental & Earth Sciences. 7(12):1–17.
DOI: https://doi.org/10.30564/jees.v8i1.11970

integrated strategy merges the performance enhancement offered by advanced co-doping, such as extended visible-light absorption and suppressed charge recombination, with the core principles of green chemistry and circular economy. It offers a dual benefit: creating highly effective photocatalysts for the degradation of persistent pollutants and valorizing abundant agricultural or biological waste streams. Our comprehensive evaluation goes beyond description to critically assess the underlying mechanisms, comparative efficacy, scalability challenges, and future research directions of this emerging field. This review underscores the unique contribution of biomass-mediated synthesis to advancing sustainable nanotechnology for environmental applications.

Keywords: Green Synthesis; Photocatalyst; Degradation; Azo Dyes; Doping; Co-Doping

1. Introduction

The textile, cosmetic, and printing industries have expanded rapidly, resulting in the release of synthetic organic dyes into aquatic habitats globally. This is resulting in a significant environmental issue. Azo dyes, characterized by one or more azo (-N=N-) bonds connected to aromatic systems, are the predominant category of commercial dyes, accounting for 60–70% of all dyes utilized[1,2]. Their intricate aromatic structure imparts a profound colouration and renders them highly resistant to natural degradation, explaining their persistence in aquatic environments.

The environmental impact is severely detrimental. The discharge of dye-contaminated effluents into aquatic environments impedes light penetration, adversely affecting photosynthesis and disrupting aquatic food webs[3]. Significantly, numerous azo dyes and the molecules that decompose them, particularly aromatic amines produced under anaerobic conditions, are toxic, induce mutations, and are carcinogenic. They can directly jeopardize human health when they contaminate food and water[4,5].

Conventional techniques for wastewater treatment, including adsorption, coagulation, and flocculation, have proven ineffective. Although adsorption is user-friendly, it frequently transfers pollutants from water to a solid phase, creating a secondary waste issue without decomposing the dye molecules[6]. This significant issue underscores the urgent necessity for improved treatment technologies capable of fully mineralizing these persistent pollutants. Among these, semiconductor photocatalysis has emerged as a premier destructive and enduring solution. Consequently, advanced oxidation processes, particularly semiconductor photocatalysis, have emerged as destructive and sustainable alternatives capable of mineralizing dyes into harmless end products like CO_2 and H_2O[1,7,8]. This review focuses on ZnO-based photocatalysts, examining their limitations and the strategies to overcome them, with a novel emphasis on the convergent approach of biomass-mediated in-situ P-N co-doping.

Due to these advantageous features, azo dyes are widely applied in multiple sectors, including the textile industry, which is the largest consumer, as well as in cosmetics, food packaging, plastics, leather, and pharmaceutical formulations. Their versatility has also enabled widespread use in laboratory settings, where they function as pH indicators, biological stains, and model pollutants for wastewater treatment studies. Prominent examples include Methyl Orange, Methyl Red, Methyl Blue (MB), and Rhodamine B. These compounds are often incorporated into consumer goods and experimental systems owing to their well-characterized optical properties and chemical stability. Figure 1 illustrates some of the common industrial and cosmetic applications of organic azo dyes, underscoring their integration into everyday products and processes[3,7,8].

Environmental Science Sustainability

Despite their functional importance, the environmental implications of azo dye release are increasingly alarming. Effluents from textile and related industries often contain high concentrations of these dyes, which are discharged into aquatic environments without adequate treatment. Due to their synthetic origin, aromaticity, and complex molecular structures, azo dyes are highly resistant to natural biodegradation pathways. They tend to persist in water bodies, adsorb onto sediments, and accumulate in aquatic organisms, creating long-term ecological risks[8–10]. Their strong coloration reduces light penetration in water, thereby impairing photosynthetic activity in aquatic flora and destabilizing entire ecosystems[11–15].

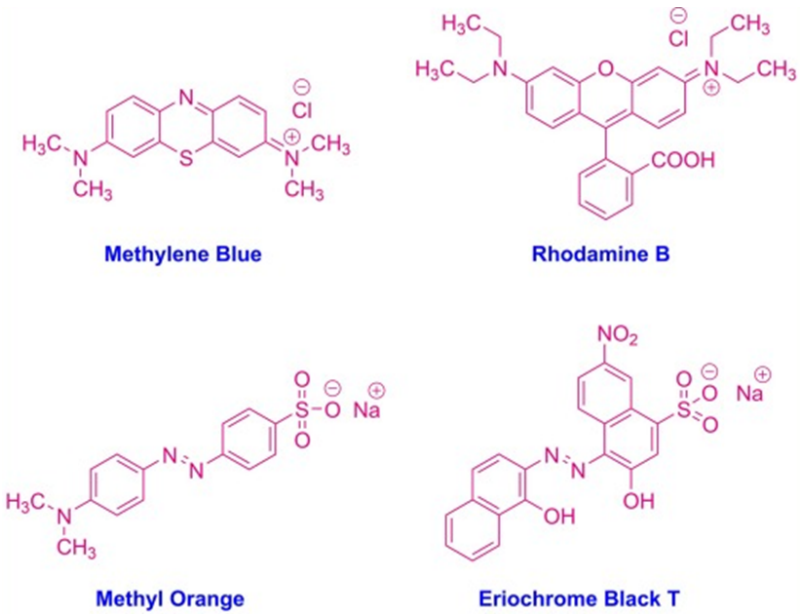

Figure 1. Various types of dyes and chemical structures [6].

More critically, the toxicity of azo dyes and their breakdown products pose direct threats to human and animal health. Upon reductive cleavage in anaerobic conditions, azo dyes can release aromatic amines, many of which are mutagenic or carcinogenic. Chronic exposure to these compounds has been linked to skin irritation, respiratory issues, and increased cancer risks. Furthermore, the recalcitrant nature of these pollutants renders conventional treatment methods such as coagulation, flocculation, and activated sludge largely ineffective for their complete removal. Even adsorption-based approaches, though promising, often suffer from cost and regeneration limitations. Given these challenges, there is an urgent need for innovative and sustainable water treatment strategies tailored specifically for azo dye degradation. Recent advancements in nanotechnology, green chemistry, and photocatalysis offer promising avenues for developing cost-effective and eco-friendly solutions. Addressing the persistence and toxicity of azo dyes through such approaches is critical for safeguarding environmental quality and public health [3,8,9].

The widespread discharge of untreated dye-laden effluents remains a pressing environmental and public health concern, particularly in regions where wastewater management infrastructure is insufficient or poorly regulated. While some textile manufacturers employ wastewater treatment systems to break down harmful azo dyes before discharge, a significant proportion of effluents are released directly into rivers, lakes, and streams without any form of remediation. This practice poses severe ecotoxicological risks, with both immediate and long-term consequences for aquatic ecosystems, agriculture, and human health [16–19].

In developing countries, where regulatory oversight and treatment facilities are often limited, contaminated water is frequently diverted for agricultural irrigation. This introduces dyes into soil systems, leading to altered physicochemical properties, reduced microbial diversity, and impaired soil fertility. Several studies have reported decreases in seed germination rates and crop productivity when irrigated with dye-contaminated water, raising serious concerns about food security and agricultural sustainability. Once released into aquatic environments, azo dyes further exacerbate ecological disturbances by blocking sunlight penetration. This reduction in light availability inhibits photosynthesis in algae and aquatic plants, disrupting primary production and destabilizing entire aquatic food webs [13,19–22].

The bioaccumulation of dyes and their metabolites in aquatic organisms presents additional ecological and toxicological risks. Fish and other aquatic species ingest dissolved dyes or contaminated sediments, leading to the metabolic breakdown of these compounds into hazardous intermediates. Such bio-transformed products can impair the growth, reproduction, and survival of aquatic organisms while also threatening higher trophic levels, including birds and humans, through biomagnification. In humans, exposure to azo dyes may occur through ingestion of contaminated water

and food, or via direct dermal contact in occupational and consumer settings. These dyes and their breakdown products, particularly aromatic amines, have been associated with a range of adverse health outcomes, including allergic reactions, oxidative stress, tumour formation, organ toxicity, and even stroke (see **Figure 2**)[23,24]. Furthermore, the human gut microbiota plays a critical role in converting azo dyes into carcinogenic aromatic amines, which can accumulate in organs such as the liver, kidneys, and bladder, significantly elevating long-term health risks[25–27].

Figure 2. Common industrial and cosmetic applications of organic azo dyes[10].

Efforts to mitigate the risks posed by azo dyes have led to the development of various treatment technologies (see **Figure 3**). Conventional physical and chemical techniques, including coagulation, flocculation, oxidation, and adsorption, have been widely employed to remove dyes such as Methyl Red (MR), Methylene Blue (MB), Methyl Orange (MO), and Rhodamine B (RhB) from wastewater. Among these, adsorption remains the most applied method due to its operational simplicity and relatively high efficiency at low concentrations[1,7,8]. However, adsorption alone does not completely degrade dyes; instead, it transfers contaminants from one phase to another, creating secondary disposal challenges. Moreover, the high cost of adsorbent regeneration, limited selectivity, and reduced efficiency at high pollutant loads significantly limit its scalability for industrial applications.

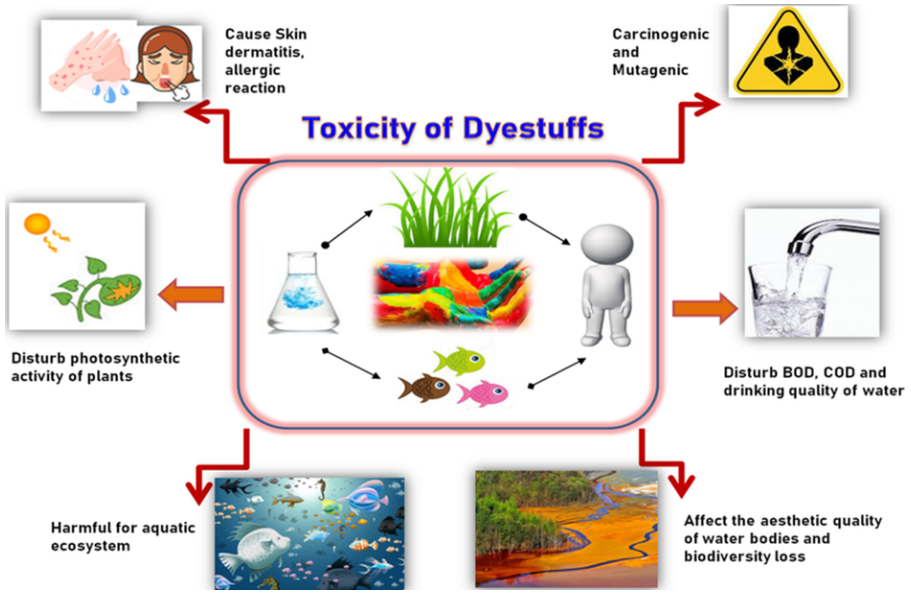

Figure 3. Health and environmental risks associated with azo dyes[18].

To overcome these limitations, researchers have increasingly turned to advanced oxidation processes, particularly photocatalysis, as a sustainable alternative [8–10]. Photocatalytic treatment involves the use of semiconductor catalysts that, when activated by ultraviolet (UV) or visible light, generate electron-hole pairs capable of producing reactive oxygen species (ROS). These highly reactive species can effectively attack dye molecules, breaking down their complex aromatic structures and mineralizing them into harmless end products such as carbon dioxide, water, and inorganic ions. Unlike adsorption, photocatalysis offers the advantage of complete pollutant degradation rather than simple phase transfer. Additionally, it operates under ambient conditions, requires minimal chemical input, and can be adapted to use solar energy, making it attractive for deployment in resource-constrained regions. This light-driven mechanism, while discussed here in the context of organic dye degradation, is a versatile advanced oxidation process also effectively applied for the remediation of other persistent pollutants, such as heavy metal ions [28].

Despite significant progress, the large-scale application of photocatalysis still faces challenges related to catalyst efficiency, recovery, and stability. Current research is therefore focused on the development of green-synthesized photocatalysts, often derived from biomass waste, to enhance performance while reducing cost and environmental footprint. Such innovations offer a dual advantage: mitigating the risks associated with azo dye pollution and simultaneously promoting sustainable waste valorisation strategies. By bridging ecological safety with technological advancement, photocatalytic treatment represents a promising pathway for addressing one of the most persistent environmental challenges of the textile and dye industries [8].

2. Photocatalysis: Principles and the Promise of ZnO

Photocatalysis is a surface-mediated process driven by the photoexcitation of a semiconductor. Upon absorbing a photon with energy ($h\nu$) ≥ Eg (the bandgap energy), an electron (e^-) is promoted from the valence band (VB) to the conduction band (CB), creating a charge-separated state, an electron-hole (e^-/h^+) pair. The efficacy of the process depends on the competition between the migration of these charge carriers to the surface to drive redox reactions and their bulk recombination, which releases energy as heat or light.

The photocatalytic activity is governed by the following key surface reactions:

Hole-mediated oxidation: The photogenerated hole (h^+), a powerful oxidant (EVB ~ +2.5 to +3.0 V for ZnO), can directly oxidize organic adsorbates or, more commonly, react with surface-bound water or hydroxide ions (OH^-) to generate hydroxyl radicals (•OH), one of the most potent oxidizing species known (E° = +2.8 V).

$$h^+ + H_2O \rightarrow \bullet OH + H^+$$

$$h^+ + OH^- \rightarrow \bullet OH$$

Electron-mediated reduction: The photogenerated electron (e^-) in the conduction band (ECB ~ –0.5 to −0.1 V for ZnO) can reduce adsorbed molecular oxygen (O_2) to yield superoxide anion radicals ($O_2\bullet^-$).

$$e^- + O_2 \rightarrow O_2\bullet^-$$

These $O_2\bullet^-$ species can further protonate to form hydroperoxyl radicals ($HO_2\bullet$) and hydrogen peroxide (H_2O_2), which ultimately lead to additional •OH generation. It is this cascade of reactive oxygen species (ROS), particularly the •OH radical, that non-selectively attacks and mineralizes the complex aromatic structures of azo dyes. These species are potent oxidizing agents that attack and mineralize complex organic molecules into carbon dioxide, water, and inorganic ions [3,7].

In dye degradation applications, the photocatalytic process relies heavily on a catalyst's ability to (i) absorb light efficiently, (ii) generate and separate electron–hole pairs, and (iii) provide active redox sites for ROS formation. Nanomaterials are especially advantageous in this regard due to their high surface-to-volume ratios and size-dependent optical properties, which can be engineered to enhance photocatalytic activity [10]. Unlike adsorption, which only transfers pollutants from one phase to another, photocatalysis enables chemical breakdown at the molecular level, offering the possibility of complete pollutant destruction.

Zinc oxide (ZnO) is among the most studied photocatalysts due to its high exciton binding energy, strong oxidative capability, low cost, and non-toxic nature [8]. However, pure ZnO faces three major challenges in practical applications:

Its relatively wide bandgap (~3.37 eV) restricts activation to the ultraviolet (UV) region, limiting solar utilization.

Rapid recombination of photogenerated electron-hole pairs lowers quantum efficiency; and

Susceptibility to photo-corrosion reduces long-term stability[9].

These limitations have driven extensive research into modifying ZnO to improve charge separation, extend light absorption into the visible region, and enhance overall photocatalytic performance. In addition, these challenges necessitate advanced material engineering strategies, such as doping and co-doping.

Photocatalytic Degradation

Although photocatalysis aims for complete mineralization of dyes, achieving this ideal outcome is often challenging. Degradation typically proceeds through several intermediates, whose formation and persistence depend on factors such as catalyst efficiency, dye structure, and reaction conditions. Notably, some intermediates, such as aromatic amines, produced during the cleavage of azo dyes may be more toxic or persistent than the original dye molecule[24]. Therefore, it is crucial to monitor degradation pathways using techniques like liquid chromatography–mass spectrometry (LC-MS) to ensure that photocatalysis does not generate harmful secondary pollutants. Optimizing photocatalytic systems thus requires not only improving ROS generation and charge-carrier dynamics but also designing reaction conditions that minimize intermediate accumulation and promote progression toward full mineralization.

3. Engineering ZnO for Enhanced Performance: The Role of Doping and Co-Doping

Doping is a widely employed strategy to overcome the intrinsic limitations of pure ZnO, including its wide bandgap, rapid electron hole recombination, and susceptibility to photo corrosion. By introducing foreign atoms into the ZnO crystal lattice, doping alters its electronic structure, surface states, and optical properties. This modification can create new energy levels within the bandgap, allowing absorption of lower-energy photons and extending ZnO's activity into the visible-light spectrum[11,12]. Single-element doping, whether with non-metals such as nitrogen (N), carbon (C), and phosphorus (P), or metals such as cobalt (Co), manganese (Mn), and magnesium (Mg), has been shown to narrow the bandgap and improve charge separation to varying degrees. Non-metal doping introduces localized states above the valence band (VB), particularly in the case of N and P, which can reduce the effective bandgap and enhance visible-light absorption[15,16]. Metal-ion doping often induces lattice distortions and modifies optical properties, typically producing a hyperchromic shift associated with enhanced visible-light absorption. However, single element doping alone cannot fully resolve the combined challenges of rapid charge recombination and long-term photo corrosion.

For this reason, co-doping simultaneous incorporation of two or more dopants has emerged as a more effective and synergistic approach. Numerous studies have shown that co-doped ZnO systems outperform their singly doped counterparts by enabling better bandgap tuning, improved charge-carrier dynamics, and enhanced structural stability[13,14]. Among the various co-doping strategies, phosphorus–nitrogen (P-N) co-doping is particularly promising due to the complementary electronic interactions of these two elements within the ZnO lattice. Individually, nitrogen typically substitutes for oxygen atoms, forming acceptor states (N 2_p) just above the VB. This narrows the bandgap but may also create recombination centres. Phosphorus, due to its larger atomic radius, preferentially replaces zinc atoms and introduces donor states below the conduction band (CB), while also generating oxygen vacancies and localized lattice distortions. When incorporated simultaneously, P and N form a cooperative "donor–acceptor pair," establishing a persistent intermediate energy band between the VB and CB. This intermediate band acts as a "stepping stone" that enables a two-step photon absorption process (VB → intermediate band → CB) under visible-light irradiation with significantly lower energy requirements[15,16].

Additionally, the P-N donor–acceptor pair produces an internal electric field that spatially separates photogenerated electrons and holes, substantially suppressing recombination and enhancing photocatalytic efficiency. The charge compensation between P^{3-} and N^{3-} also improves lattice stability, reducing photo corrosion and further strengthening the catalyst's durability. Through these combined effects, P–N co-doping achieves simultaneous bandgap narrowing, enhanced charge separation pathways, and improved struc-

tural integrity advantages that cannot be fully realized by single-element doping strategies alone [15,16,24]. **Table 1** provides a comparative analysis of single (N or P) versus P-N co-doping effects on ZnO's photocatalytic properties.

Table 1. Comparative analysis of doping strategies for ZnO photocatalysts.

Doping Type	Bandgap Modification	Charge Separation	Visible Light Activity	Stability	Key Challenges
Undoped ZnO	Wide (~3.37 eV)	Poor	Low	Moderate	UV-only, fast recombination
N-doping	Reduced	Slightly Improved	Moderate	Can create recombination canters	Limited performance gain
P-doping	Reduced	Improved	Moderate	May induce lattice strain	Often requires harsh precursors
P-N Co-doping	Significantly Reduced & Creates Intermediate Band	Greatly Enhanced (Internal Field)	High	Improved (Charge compensation)	Synthesis control, dopant uniformity

4. Green Synthesis: Merging Sustainability with Performance

Material engineering enhances performance; yet conventional methods for synthesizing these nanomaterials employ toxic chemicals, require substantial energy, and generate hazardous by-products, adversely impacting the environment. This contradicts the concept of developing "green" solutions for environmental remediation. Consequently, the research is increasingly advancing towards sustainable synthesis methodologies [17]. Green synthesis, particularly utilizing plant biomass extracts or agricultural waste, is a more environmentally favourable option. Plant extracts are rich in bioactive chemicals, including polyphenols, flavonoids, and alkaloids, which facilitate the formation of nanoparticles by serving as natural reducing, capping, and stabilizing agents [18]. This indicates that hazardous substances are no longer required.

The novel concept central to this review is the use of biomass not just as a reducing agent, but as an in-situ source of dopants. For instance, banana peels are rich in potassium, phosphorus, and nitrogen compounds. Utilizing such biomass allows for the simultaneous green synthesis of ZnO and the incorporation of P and N into its lattice during formation [19]. This novel approach integrates the pursuit of enhanced photocatalytic activity with principles of green chemistry and the utilization of agricultural waste to create valuable products. It represents a dramatic transition from using biomass solely as a "green" reducing agent to exploiting it as a multifaceted precursor for simultaneous nanoparticle synthesis and electronic structure alteration. This represents a significant advance over sequential processes, reducing steps, waste, and the need for external, potentially hazardous dopant chemicals.

5. Synthesis and Morphology of ZnO Nanostructures

Nanoparticles can be synthesized using a variety of techniques, broadly categorized into top-down and bottom-up approaches. Top-down methods such as electrochemical oxidation, arc discharge, and laser ablation involve breaking down bulk carbonaceous materials into nanoscale particles through physical exfoliation processes. While effective, these methods often require harsh experimental conditions, involve complex procedures, and rely on expensive equipment and chemicals. These limitations restrict their practicality for large-scale or routine applications (see **Figure 4**) [13,22–24].

In contrast, bottom-up approaches, including microwave-assisted synthesis, solvothermal, ultrasonic, and pyrolysis methods, build nanoparticles from smaller molecular precursors through processes such as carbonization and passivation [22]. These methods are generally more cost-effective, easier to operate, and require simpler equipment and reagents. As a result, they have become widely adopted for nanoparticle synthesis.

The synthesis of ZnO nanoparticles (ZnO NPs) can be achieved through both conventional and green methods, with each technique influencing the resulting particle's properties. Green synthesis methods have gained attention for their environmental friendliness, simplicity, and use of natural reducing agents, offering a sustainable alternative to conventional chemical routes. Of more interest in this review is a sol-gel method.

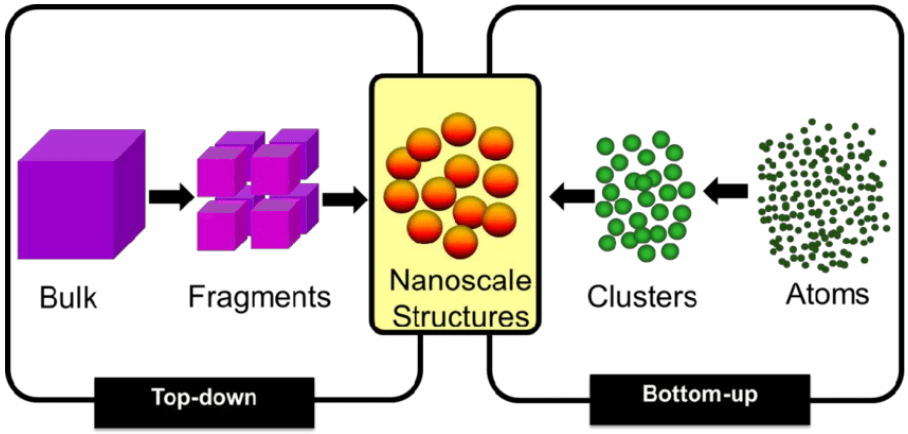

Figure 4. Top-down and Bottom-up approach for nanoparticles synthesis [23].

The choice of synthesis method is paramount, as it dictates the key physicochemical properties of ZnO nanoparticles that govern photocatalytic efficiency, namely, crystallinity, particle size and morphology, surface area, and defect density.

5.1. Sol-Gel Process

The sol-gel method is prized for its excellent control over stoichiometry and its suitability for producing thin films, which are advantageous for catalyst recovery [25]. However, the need for a high-temperature calcination step to achieve crystallinity often leads to particle agglomeration, reducing the effective surface area and potentially masking active sites.

The sol-gel method is a wet-chemical synthesis route involving the transition of a liquid "sol" into a solid "gel" through hydrolysis and condensation reactions (see **Figure 5**). In ZnO synthesis, typical precursors include zinc acetate dihydrate**,** zinc nitrate**,** or zinc chloride**,** which are dissolved in solvents such as ethanol, methanol, or water. The process generally begins with dissolving the precursor in a suitable solvent to form a homogeneous solution. A base such as sodium hydroxide (NaOH), potassium hydroxide (KOH), or ammonia is then introduced to initiate hydrolysis. During hydrolysis, water molecules react with the zinc precursor to form zinc hydroxide species. These then undergo condensation reactions, producing Zn-O-Zn linkages that gradually lead to the formation of a sol, a colloidal suspension of ZnO nanoparticles. As the reaction continues, the sol evolves into a gel, a three-dimensional solid network with solvent trapped in its pores. This gelation marks a critical transition where particle aggregation and structural formation occur [11,15,20,26,29–33].

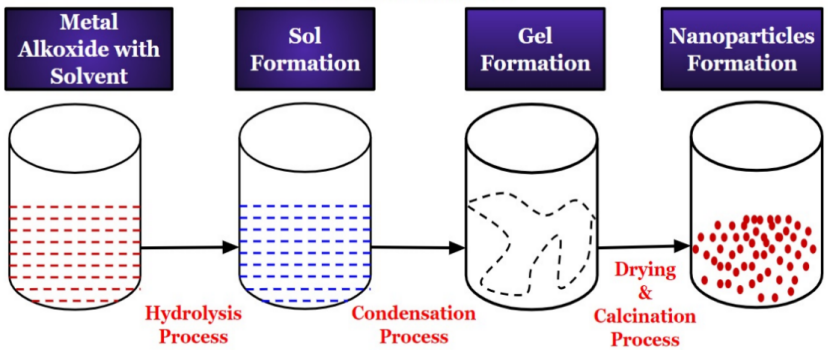

Figure 5. Sol-gel method for ZnO NP's synthesis [25].

Following gelation, the material undergoes aging, which allows further polymerization and network stabilization. The gel is then subjected to drying, typically at room temperature or under mild heating, to remove solvents. This produces a xerogel (dried gel), which is then calcined at elevated temperatures (usually 300–600 °C). Calcination improves the

crystallinity of the ZnO particles, removes residual organics, and stabilizes the structure. One of the main advantages of the sol-gel process is the fine control over product properties such as particle size, porosity, and crystallinity. By adjusting synthesis parameters like precursor concentration, pH, reaction temperature, and calcination conditions, researchers can tailor ZnO structures to meet specific application needs. Additionally, the process is compatible with thin film deposition techniques like spin-coating and dip-coating, making it suitable for fabricating ZnO films for electronic and optoelectronic applications. The sol-gel method is also considered environmentally friendly and energy-efficient due to its relatively low processing temperatures and minimal equipment requirements (**Figure 5**)[29–31]. However, it does require careful control of synthesis conditions to avoid issues such as cracking, shrinkage, or agglomeration during drying and calcination[32–34].

The morphology of semiconductor nanoparticles is a critical factor governing their photocatalytic efficiency, as it directly influences surface area, active site availability, and charge carrier dynamics. Electron microscopy (SEM and TEM) analysis provides vital insights into these structural characteristics across different synthesis routes.

As evidenced in **Figure 6**, ZnO nanoparticles synthesized via the sol-gel method form dense, spherical micro-agglomerates. This is a typical outcome of the sol-gel process, where the condensation of Zn-O-Zn linkages during gelation leads to the assembly of primary particles into larger clusters. The accompanying TEM image confirms that these agglomerates are composed of primary nanoparticles with a near-spherical morphology and an average size of 30–35 nm. While this agglomeration can slightly reduce the effective surface area, the sol-gel method consistently produces particles with high purity and crystallinity after calcination, which is beneficial for photocatalytic activity.

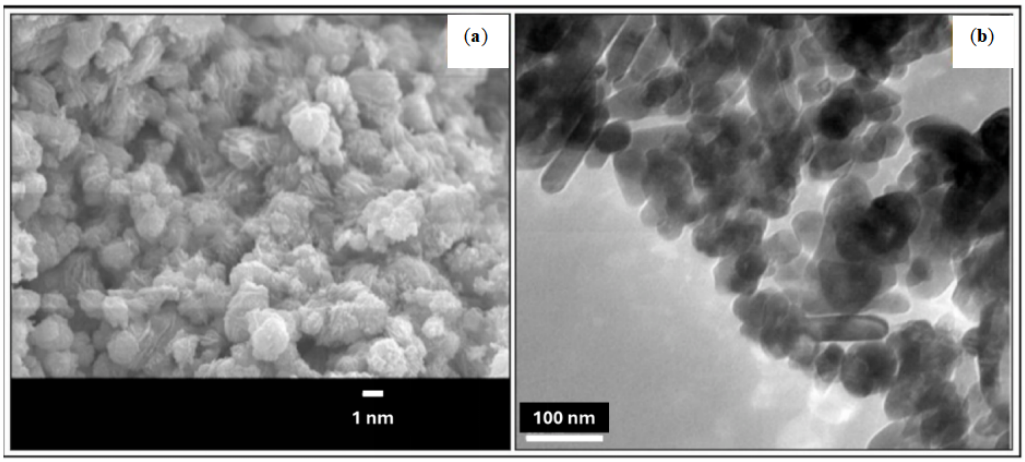

Figure 6. (**a**) SEM image of ZnO NP's; (**b**) TEM image of ZnO NP's synthesized by Sol-gel[11].

5.2. Precipitation Method

The precipitation method is arguably the most cost-effective and scalable route. Its simplicity, however, comes at the expense of precise control. As evidenced in **Figure 7**, the method can produce uniform primary particles (e.g., spindle-shaped NPs), but these often form larger, ill-defined agglomerates. The use of surfactants can improve morphology but adds complexity and cost, negating one of its primary advantages. A simple yet affordable technique, precipitation is often employed in large-scale and mass production (**Figure 8**). To change the morphology, basic equipment, appropriate surfactants, and doping metals are required. ZnO NPs made using the precipitation process have a surface morphology that is like a nanosphere with a few nanometres in diameter.

In contrast, the precipitation method (**Figure 7**) yields a more defined and uniform primary structure. The TEM analysis reveals well-dispersed, spindle-shaped nanoparticles with an average diameter of 25 nm. This anisotropic shape is advantageous as it can provide a higher surface-to-volume ratio compared to spherical particles and may favour directional charge transfer. The SEM image, showing larger spherical assemblies, highlights that these primary spindle-shaped particles can still form secondary structures, but the fundamental building blocks are highly uniform, suggesting a controlled nucleation and growth phase during precipitation.

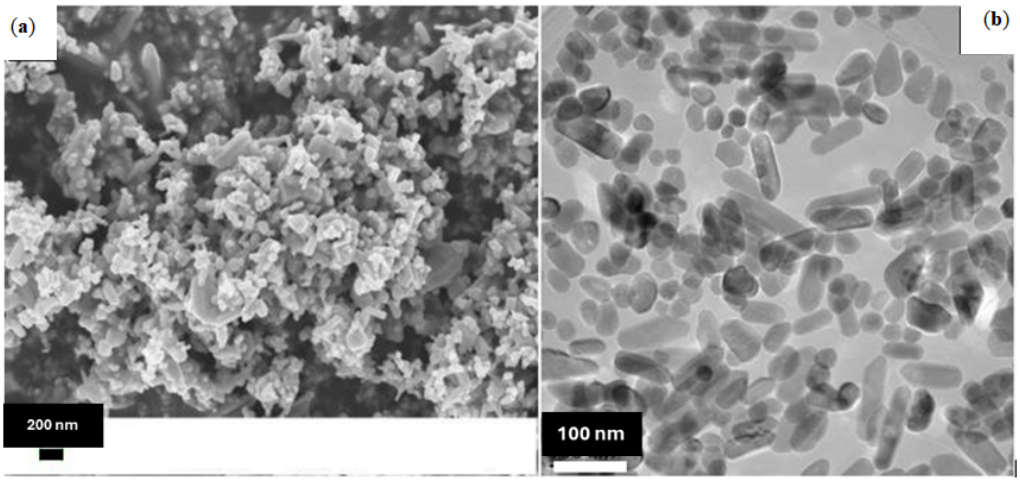

Figure 7. (**a**) SEM image and (**b**) TEM image of ZnO NP's synthesized by Precipitation method [11].

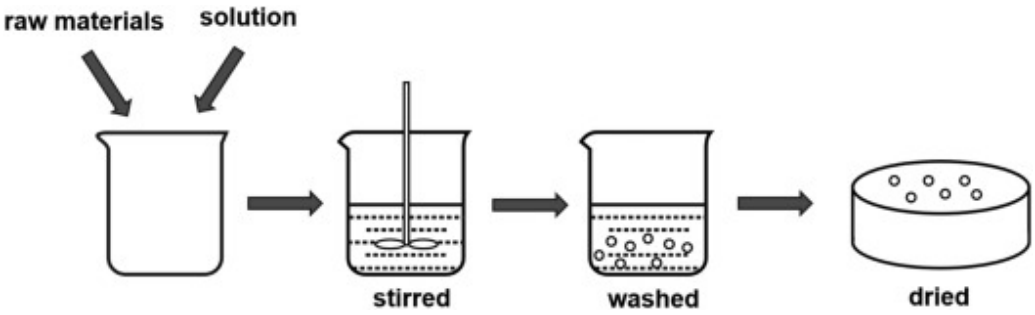

Figure 8. Precipitation method for ZnO NP's synthesis [25].

5.3. Microwave Assisted Method

The microwave-assisted synthesis of ZnO nanoparticles (ZnO NPs) offers a rapid, clean, and energy-efficient route, with no need for elevated reaction temperatures. This method enables precise control over particle size and shape due to its uniform and fast heating capabilities. By adjusting the microwave power and irradiation duration, various ZnO morphologies can be obtained (see **Figure 9**). Researchers have successfully employed this technique to synthesize ultrafine ZnO particles with controlled structures. The resulting nanoparticle size and shape are influenced by factors such as the type of precursor used, irradiation time, and microwave intensity, making this approach highly tuneable for specific applications.

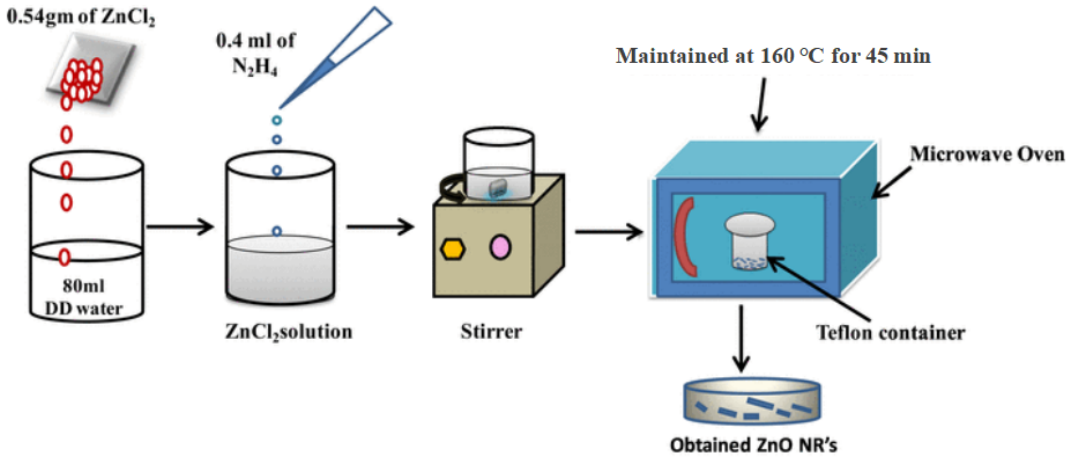

Figure 9. Microwave-assisted method for ZnO NP's [27].

The versatility of synthesis in tailoring morphology is best demonstrated by the microwave-assisted method (**Figure 10**). By simply altering precursors and irradiation parameters, this technique can produce dramatically different architectures. The microwave-assisted method represents a significant advancement in synthetic control. The ability to rapidly and uniformly heat the reaction mixture allows for the precise nucleation and growth of complex architectures, such as the flower-like or needle-like structures shown in **Figure 10**[27]. These hierarchical structures often possess very high surface areas and can facilitate better light harvesting and charge transport. The primary limitation is the translation from laboratory-scale reactors to large, continuous-flow industrial systems.

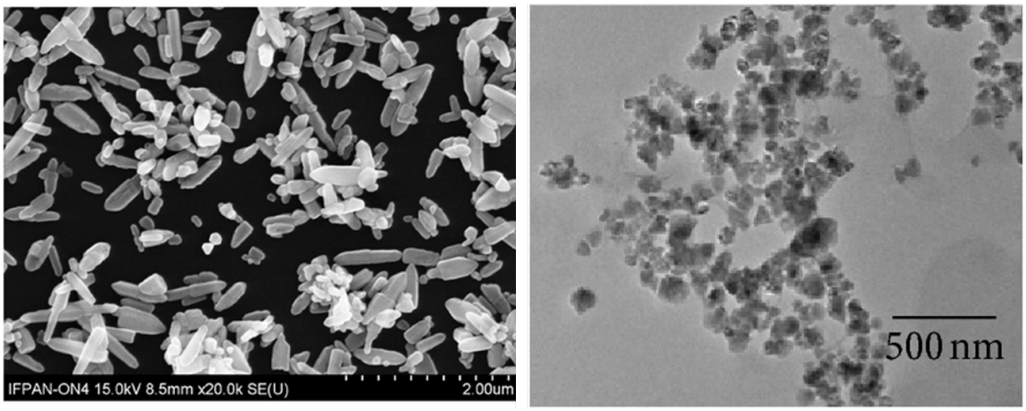

Figure 10. SEM, and TEM images of ZnO NP's synthesized by microwave-assisted method[11,35,36].

5.4. Green Synthesis

Green synthesis methods that utilize microorganisms, plant extracts, and biopolymers are increasingly recognized as sustainable and eco-friendly alternatives to conventional chemical and physical routes for nanomaterial production (**Figure 11**). Traditional synthesis techniques often depend on toxic chemicals, high temperatures, or costly reagents, which pose environmental and health risks. In contrast, green synthesis aligns with the principles of green chemistry by minimizing hazardous by-products, reducing energy requirements, and making use of renewable biological resources.

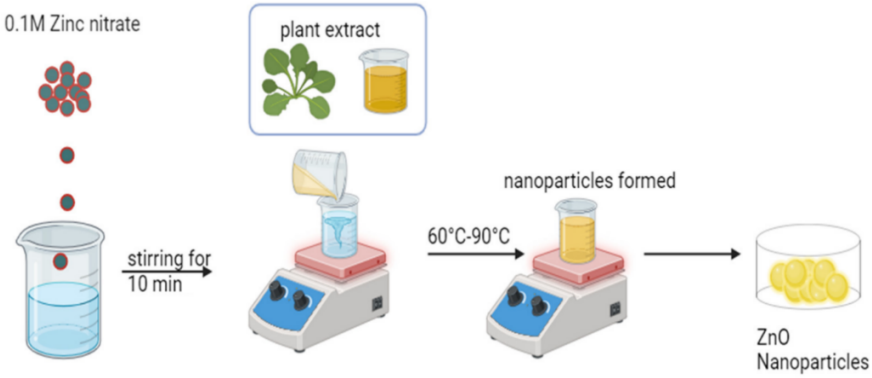

Figure 11. Green method for the synthesis of ZnO NP's[37].

Among the various green approaches, plant-based synthesis has emerged as one of the most promising due to the accessibility, affordability, and phytochemical richness of plant materials. Plant extracts contain abundant secondary metabolites, including flavonoids, tannins, phenolic acids, terpenoids, saponins, and alkaloids, which serve as natural reducing, capping, and stabilizing agents during nanoparticle formation. For example, phenolics and flavonoids donate electrons to reduce metal ions, while tannins and alkaloids help stabilize nanoparticles and prevent agglomeration. This

intrinsic multifunctionality removes the need for additional synthetic reducing or capping agents, making the overall process more environmentally benign [1–5].

As highlighted in **Table 2**, green synthesis occupies a distinctive niche in nanomaterial production. While its ability to precisely control nanoparticle size, morphology, and crystallinity is generally lower than that of advanced physical or microwave-assisted methods, its unique advantages lie in sustainability, low toxicity, and the multifunctional role of biomolecules. Plant-based systems not only drive nanoparticle reduction but also provide inherent functionalization and even in situ doping. Natural polyphenols, for instance, can passivate surface defects and potentially reduce charge-recombination centres, contributing to enhanced photocatalytic or optical properties [19].

However, a key challenge remains the intrinsic variability of biological materials. Differences in plant species, growing conditions, extraction methods, and phytochemical composition can affect nanoparticle yield, morphology, and functional properties. This variability makes reproducibility and mechanistic understanding more difficult compared to well-defined chemical synthesis routes. Nonetheless, the sustainability and multifunctionality of green synthesis continue to make it an attractive and rapidly expanding area in nanomaterials research.

ZnO nanoparticles synthesized using plant extracts have attracted significant attention due to their multifunctional properties. Studies have demonstrated that green-synthesized ZnO nanoparticles exhibit excellent antibacterial activity against a wide range of pathogens. This is attributed to their ability to generate reactive oxygen species (ROS), disrupt microbial cell membranes, and release $Zn2^{+}$ ions, which collectively inhibit bacterial growth. Furthermore, the photocatalytic efficiency of ZnO nanoparticles produced through plant-based synthesis has been reported to be superior to those prepared by conventional methods. When exposed to UV or visible light, these nanoparticles effectively degrade various organic pollutants, including dyes and pharmaceuticals, making them highly suitable for wastewater treatment applications. Beyond environmental remediation, green-synthesized ZnO nanoparticles also hold promise in biomedical fields. Their biocompatibility and lower toxicity compared to chemically synthesized counterparts make them attractive for drug delivery, wound healing, and anticancer therapies. Overall, the integration of plant-based green synthesis with nanotechnology provides a sustainable pathway for producing functional nanomaterials while addressing pressing environmental and healthcare challenges [3–5].

Table 2. Critical comparison of ZnO nanoparticle synthesis methods for photocatalytic applications.

Synthesis Method	Typical Morphology	Control Over Size	Crystallinity	Typical Surface Area (m^2/g)	Scalability & Cost	Environmental & Practical Considerations
Sol-Gel	Spherical agglomerates, thin films	Moderate (Good for films, less for discrete particles)	High (after calcination)	10–50	Moderate scalability; Low-to-moderate cost	Requires calcination; Can use benign precursors but may involve organic solvents.
Precipitation	Spherical clusters, spindle-shaped particles	Moderate to Good (influenced by surfactants)	Moderate to High	20–60	Highly scalable; Very low cost	Aqueous, simple process; Potential for anion impurities.
Microwave-Assisted	Nanosheets, needles, flowers	Excellent (Highly tunable via power/precursors)	High	30–100+	Moderate scalability; Moderate cost (energy)	Rapid, energy-efficient; Reproducibility can be equipment-dependent.
Green/Biomass-Mediated	Spherical, hexagonal, highly agglomerated	Low to Moderate (Biomass chemistry dictates morphology)	Variable (often high)	15–50	Highly scalable; Very low cost (uses waste)	Most sustainable; Introduces natural dopants; Hard to precisely control.

One of the most important characterizations of ZnO nanoparticles is their morphology, as it provides valuable insight into their structural and functional properties. Scanning Electron Microscopy (SEM) analysis revealed that the nanoparticles generally possess a semi-spherical shape and tend to form highly agglomerated clusters, which is often attributed to their large surface energy. To obtain a more detailed understanding of the particle size and shape, Transmission Electron Microscopy (TEM) was employed. As shown in **Figure 12b**, TEM micrographs confirmed the presence of well-defined particles exhibiting shapes that ranged from nearly spherical to distinctly hexagonal. The measured average grain size was approximately 35.5 nm, which aligns with earlier findings reported in the literature. Such morphological features strongly influence the optical, electronic, and photocatalytic properties of ZnO nanoparticles, making their characterization essential for environmental and biomedical applications [26].

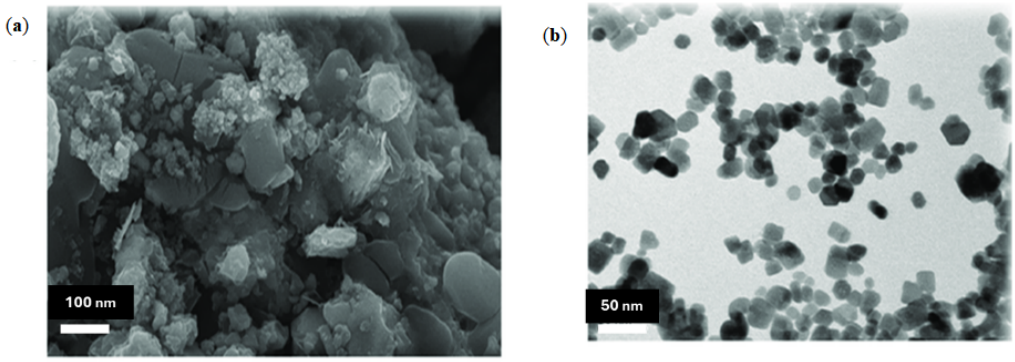

Figure 12. (**a**) SEM; and (**b**) TEM magnifications of ZnO NP'S synthesized by green method [37].

6. Degradation Comparison of Materials Including ZnO and Other Catalysts

Table 3 consolidates and critically compares the degradation efficiency of ZnO against other metal oxides (TiO_2, SnO_2) for common azo dyes. The table highlights comparative studies where ZnO consistently outperforms other widely investigated metal oxides, such as TiO_2 and SnO_2, in terms of both degradation percentage and shorter reaction times. For instance, ZnO achieves nearly complete removal of Methyl Orange (99.7%) within just 30 min, while TiO_2 requires 180 min to reach only 87%. Similarly, for Methylene Blue and Rhodamine B, ZnO shows higher degradation rates in shorter time intervals compared to its counterparts. All the degradation reactions were conducted under UV light, apart from Rhodamine B using TiO_2 which was conducted under sunlight. These findings clearly demonstrate ZnO's superior catalytic performance, making it a highly promising and cost-effective material for large-scale environmental remediation, especially in addressing dye-contaminated industrial effluents.

Table 3. Applications of metal oxides for degradation of organic azo dyes [11].

Pollutant	Metal Oxide	Catalyst Mass (mg)	Dye Conc. (mg/L)	% Degradation (%)	Time (min)	Ref.
Methylene Blue	ZnO	10	20	92.5	180	Balcha et al. [38]
	TiO_2	10	100	90.0	180	Nguyen-Phan and Shin [39]
	SnO_2	30	10	90.7	180	Sadeghzadeh-Attar [40]
Methyl Orange	ZnO	10	30	99.7	30	Chen et al. [41]
	TiO_2	10	20	87.2	180	Subha and Jayaraj [42]
	SnO_2	60	10	97.4	120	John et al. [43]
Rhodamine B	ZnO	150	10	95.1	70	Rahman et al. [44]
	TiO_2	100	20	92.4	180	Barkul et al. [45]
	SnO_2	50	10	82.1	60	Ji et al. [46]

7. The Convergence: Biomass-Mediated P-N Co-Doping for Dye Degradation

This section constitutes the core novel contribution of the review. We move beyond describing separate concepts to analytically converge them. The integration of biomass-mediated green synthesis with intentional P-N co-doping addresses multiple sustainability pillars:

- Environmental: Degrades persistent pollutants.
- Economic: Valorizes low-cost agricultural waste.
- Social: Promotes safer, greener nanomaterial production.

Mechanistic Insight: The process leverages biomass (e.g., fruit peels, plant leaves) rich in phosphorylated compounds (phospholipids, ATP) and nitrogenous molecules (proteins, amino acids, chlorophyll). During thermal or hydrothermal treatment, these biomolecules decompose, releas-

ing P and N species that incorporate into the growing ZnO lattice, creating the beneficial donor-acceptor pairs discussed earlier [20,21]. This one-pot synthesis bypasses the need for separate, toxic dopant precursors like ammonium phosphate.

Critical Analysis of State-of-the-Art: Preliminary studies are promising. For example, research using Hibiscus cannabinus extract for ZnO synthesis reported enhanced dye degradation, attributed to natural element incorporation [31]. However, the field is nascent. Most studies infer doping from performance improvements rather than providing direct, mechanistic evidence of in-situ P-N pair formation. A significant research gap is the lack of systematic studies correlating specific biomass composition, synthesis parameters, and the resulting dopant configuration within ZnO.

8. Challenges and Considerations for Real-World Application

For this technology to transition from lab to market, several hurdles must be addressed:

- Mineralization vs. Toxicity: Complete mineralization to CO_2 and water is ideal but challenging. Toxic intermediates (e.g., aromatic amines from azo cleavage) can form [24]. Future work must employ advanced analytical techniques (LC-MS, TOC analysis) to monitor degradation pathways.
- Nanoparticle Leakage & Ecotoxicity: The potential release of ZnO nanoparticles into treated effluent poses an ecotoxicological risk [26]. Strategies like catalyst immobilization on membranes, polymers, or magnetic substrates are essential for recovery and reuse.
- Life-Cycle Assessment (LCA): While "green" in synthesis, a comprehensive LCA is needed to quantify the overall environmental footprint, including energy use for biomass processing and catalyst reactivation.
- Reproducibility and Scalability: The variability of natural biomass requires standardization of pre-treatment and extraction protocols to ensure consistent photocatalyst performance at scale.

9. Conclusion and Research Gaps

This review has critically examined the innovative strategy of using biomass for the in-situ synthesis and P-N co-doping of ZnO nanoparticles for photocatalytic dye degradation. We establish that the novelty lies not in reviewing ZnO or green synthesis separately, but in synthesizing the concept of their integrated, multi-functional application where biomass serves as a simultaneous reactant, reductant, and dopant source.

To advance this promising field, future research must move from proof-of-concept to mechanistic and applied studies. We recommend the following actionable directions:

- Mechanistic Elucidation: Use advanced characterization (XPS, EPR, solid-state NMR) to definitively prove the formation and role of P-N donor-acceptor pairs within biomass-synthesized ZnO.
- Biomass Library Screening: Conduct systematic studies screening diverse N- and P-rich biomass wastes (e.g., different fruit peels, seed cakes, algae) to create a database linking biomass chemistry to photocatalytic efficacy.
- Process Optimization: Develop standardized, scalable protocols (e.g., continuous flow hydrothermal synthesis) to enhance reproducibility and control over nanoparticle properties.
- Real Wastewater Trials: Test optimized catalysts in complex, real industrial effluents containing dye mixtures, salts, and organic competitors to evaluate practical performance.
- Immobilization Engineering: Design and test robust, easily separable catalyst composites (e.g., ZnO on biochar, magnetic ZnO hybrids) to mitigate nanomaterial release and facilitate reuse.
- Sustainability Quantification: Perform thorough Life-Cycle Assessments to compare the environmental benefits of this integrated approach against conventional doping and synthesis methods.

By pursuing this integrated, critical, and solution-oriented path, researchers can develop next-generation photocatalytic technologies that are not only highly effective but also truly aligned with the principles of green chemistry and circular economy, offering a tangible solution to the global challenge of water pollution.

Author Contributions

T.E.M.: idealization, supervision, and editing. C.L.-D.: idealization, supervision, and editing. S.M.M.: supervision

and editing. T.D.M.: supervision. N.L.K.: editing. M.M.M.: resources, writing—review. All authors have read and agreed to the published version of the manuscript.

Funding

The authors received no financial support for the research, authorship, and/or publication of this article.

Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Not applicable.

Data Availability Statement

Data will be made available on request at motaungte@live.com.

Conflicts of Interest

The authors declare no conflict of interest.

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