



Synthesis and Characterization of Metal Oxide Nanoparticles for Wastewater Treatment Applications

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Abstract

Metal oxide nanoparticles are attractive materials for water and wastewater remediation because their nanoscale dimensions provide high surface area, tunable optical properties, catalytic activity and, in some systems, magnetic recoverability. This methodology paper develops a reproducible laboratory procedure for the synthesis, characterization and treatment evaluation of zinc oxide (ZnO) nanoparticles as a representative metal oxide photocatalyst. The proposed route uses a wet-chemical sol-gel/precipitation method followed by drying and calcination, after which the material is characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR) and UV-Visible spectroscopy. Wastewater-treatment performance is evaluated with a model dye solution under controlled irradiation, catalyst dosage, pH and contact time. Removal efficiency and pseudo-first-order kinetic constants are calculated from UV-Visible concentration measurements, while catalyst recovery and reusability are assessed through repeated treatment cycles. The method is grounded entirely in literature available through 2018. Pre-2019 studies show that ZnO, TiO₂ and iron-oxide nanomaterials can remove dyes, heavy metals and other pollutants through photocatalysis and adsorption [1-6]. The paper therefore provides a compact experimental framework suitable for laboratory validation, comparison of synthesis conditions and subsequent optimization for real wastewater. The results section reports literature-supported performance targets rather than claiming unperformed original measurements.

Introduction

Industrial and municipal wastewaters can contain dyes, phenolic compounds, pesticides, heavy metals and other persistent contaminants that are not always removed efficiently by conventional biological or physicochemical processes. Nanomaterials have therefore been investigated as complementary treatment materials because small particle size increases accessible surface area and can enhance adsorption, surface reaction and catalytic conversion. Lu et al. [2] reviewed the use of TiO₂, ZnO and iron oxides in water treatment and emphasized their ability to address both organic

and inorganic pollutants. At the same time, practical application requires careful control of particle synthesis, morphology, phase composition, recovery and environmental safety.

Among metal oxides, ZnO and TiO₂ are widely studied semiconductor photocatalysts. When photons with sufficient energy are absorbed, electron-hole pairs are generated and can produce reactive oxygen species that oxidize organic contaminants. ZnO is especially useful for a compact methodology study because it can be synthesized by inexpensive wet-chemical routes and characterized with common laboratory

techniques. Balcha et al. [3] directly compared precipitation and sol-gel ZnO and showed that synthesis route influences crystallite size and photocatalytic degradation. Reviews of ZnO dye treatment [7] and TiO₂ environmental photocatalysis [4] similarly show that catalyst dose, pollutant concentration, pH, irradiation and surface chemistry strongly influence treatment performance.

The objective of this paper is to present a step-by-step methodology for preparing ZnO nanoparticles, verifying their structural and optical properties, and evaluating their ability to remove a model organic pollutant from water. Comparative evidence from TiO₂ and iron-oxide studies is included to place the method within the broader metal-oxide wastewater-treatment field. All references are limited to publications from 2018 or earlier.

Literature Review

Wet-chemical synthesis is a major route for producing metal oxide nanoparticles because reaction pH, precursor concentration, temperature, solvent and additives can be adjusted to control nucleation, particle size, phase and morphology. Nikam et al. [1] reviewed precipitation, hydrothermal, sol-gel and related liquid-phase methods and noted their suitability for scalable nanoparticle manufacture. For wastewater applications, this control is important because the number and accessibility of active surface sites can determine adsorption and photocatalytic performance.

ZnO has been widely evaluated for dye degradation. Lam et al. [7] summarized ZnO photocatalytic systems and identified catalyst loading, dye concentration, pH and irradiation conditions as key operational variables. Balcha et al. [3] synthesized ZnO by precipitation and sol-gel methods, reporting wurtzite crystallite sizes of approximately 30 and 28 nm, respectively. Their sol-gel sample showed stronger methylene-blue degradation than the precipitation-derived material, demonstrating that synthesis history can alter treatment behavior. Such findings justify the use of a

controlled sol-gel/precipitation route in the present methodology.

TiO₂ provides a useful comparison because of its high photostability and extensive history in advanced oxidation. Mahlambi et al. [4] reviewed TiO₂ nanoparticle synthesis, modification and pollutant degradation, while Al-Hamdi et al. [5] reviewed SnO₂ as another metal-oxide photocatalyst for water treatment and Reddy et al. [8] discussed broader advances in photocatalytic treatment of aqueous pollutants. By 2018, modified TiO₂ systems were also being tested against complex industrial effluents: Takle et al. [9] reported vanadium-doped TiO₂ for degradation of distillery spent wash and textile dye under natural sunlight. Fulekar et al. [10] demonstrated biologically mediated TiO₂ synthesis and used the resulting material for methyl-orange photodegradation, illustrating the growing interest in greener synthesis approaches.

Not all metal oxides act mainly through photocatalysis. Magnetic iron oxides can function as adsorbents and offer convenient post-treatment separation. Lee et al. [6] prepared mixed gamma-Fe₂O₃/Fe₃O₄ nanostructures with high surface area and reported removal of arsenate, chromate and Congo red, together with magnetic recovery. This comparison is important for methodology design: photocatalytic ZnO is selected for the main experiment, while magnetic iron oxides illustrate an alternative adsorption-oriented treatment pathway.

The pre-2019 literature also emphasizes limitations. Nanoparticles can agglomerate, their recovery from treated water can be difficult, and metal-ion release or nanoparticle toxicity may complicate full-scale use. Lu et al. [2] highlighted economic, recovery and toxicity concerns, whereas the TiO₂ and ZnO literature indicates that visible-light response and recombination of photogenerated charge carriers can limit efficiency [4,7,8]. Accordingly, the present methodology includes catalyst recovery, blank controls and a reusability test rather than focusing only on single-cycle dye removal.

Table 1. Pre-2019 literature supporting the proposed methodology

Material / study	Synthesis or focus	Key wastewater-treatment relevance	Reference
ZnO	Precipitation vs sol-gel	Synthesis route affected crystallite size and methylene-blue degradation	Balcha et al. [3]
TiO ₂	Review / photocatalysis	Framework for synthesis, modification and degradation of persistent organics	Mahlambi et al. [4]
Iron oxide	Magnetic nanostructures	Adsorption of heavy metals and Congo red with magnetic recovery	Lee et al. [6]

V-TiO ₂	Sol-gel, sunlight	Applied to dye and complex distillery spent wash	Takle et al. [9]
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Methodology

1. Experimental Design and Materials

The experiment is designed around ZnO as a representative metal oxide nanoparticle, with methylene blue (MB) used as a model dissolved organic contaminant. The main factors are synthesis route, catalyst dosage, pH and irradiation time. A no-catalyst photolysis blank and a dark adsorption control are included so that pollutant loss can be separated from true photocatalytic removal. The procedure is based on the wet-chemical synthesis principles reviewed by Nikam et al. [1] and the ZnO treatment conditions reported by Balcha et al. [3] and Lam et al. [7].

Chemicals: zinc acetate dihydrate, sodium hydroxide, absolute or analytical-grade ethanol, deionized water and methylene blue.

Equipment: magnetic stirrer, centrifuge, drying oven, muffle furnace, pH meter, UV-Visible spectrophotometer, XRD instrument, SEM and FTIR spectrometer.

Recommended safety controls: gloves, eye protection, fume hood for powders/solvents, controlled nanoparticle waste collection, and avoidance of direct UV exposure.

Table 2. Suggested experimental conditions

Parameter	Recommended level	Purpose
Zinc precursor	0.1 M zinc acetate	Provides Zn ²⁺ for controlled hydrolysis
Precipitating/base agent	0.2 M NaOH; add slowly	Controls nucleation and pH
Aging time	2 h stirring + overnight aging	Promotes particle formation
Drying / calcination	100 C drying; 400-450 C, 2 h	Removes volatiles and develops crystalline ZnO
MB concentration	20 mg/L	Model wastewater concentration
Catalyst dose	0.25-1.0 g/L	Determine optimum loading

Irradiation	UV-A or controlled solar simulator	Initiates ZnO photocatalysis
Sampling	Every 20 min up to 120 min	Kinetics and removal curve

Step-by-Step Synthesis Procedure

Step 1: Prepare the precursor solution. Dissolve zinc acetate dihydrate in ethanol or a water-ethanol mixture to obtain a 0.1 M precursor. Stir until the solution becomes clear. A separate 0.2 M NaOH solution is prepared and cooled to room temperature.

Step 2: Induce hydrolysis and nucleation. Add the NaOH solution dropwise to the zinc precursor under vigorous stirring. Maintain the reaction near pH 10-11. Slow addition helps reduce uncontrolled local precipitation and promotes more uniform nucleation.

Step 3: Age the sol/precipitate. Continue stirring for approximately 2 h and then age the suspension overnight. Aging permits growth and reorganization of the zinc-containing precursor phase before isolation.

Step 4: Recover, wash and dry. Separate the solid by centrifugation. Wash repeatedly with deionized water and ethanol until residual ions and unreacted precursor are minimized. Dry the recovered solid at approximately 100 C.

Step 5: Calcine the nanopowder. Calcine the dry precursor at 400-450 C for about 2 h. This step promotes formation of crystalline wurtzite ZnO. Record the mass before and after calcination to estimate product yield. Excessive calcination should be avoided because grain growth can reduce surface area.

Step 6: Characterize the nanoparticles. Use XRD to confirm the ZnO crystal phase and estimate crystallite size with the Scherrer relationship $D = K\lambda/(\beta \cos \theta)$. SEM is used to inspect morphology and agglomeration. FTIR verifies metal-oxygen vibrations and residual surface groups. UV-Visible spectroscopy or diffuse reflectance is used to examine optical absorption and estimate the absorption edge. These techniques mirror those commonly used in the pre-2019 ZnO and TiO₂ literature [3,4,9,10].

Step 7: Prepare model wastewater and establish controls. Prepare a 20 mg/L methylene-blue

solution. For each test, place 100 mL of dye solution in a glass reactor and add the required ZnO dose. Prepare a photolysis blank without ZnO and a dark adsorption control with ZnO but no irradiation. Adjust pH with dilute acid or base and record the initial absorbance.

Step 8: Run the photocatalytic treatment. Stir the ZnO-dye suspension in the dark for 30 min to approach adsorption equilibrium, then expose it to UV-A or a controlled solar simulator. Maintain mixing to keep particles suspended. Collect aliquots every 20 min for up to 120 min. Immediately centrifuge or filter each aliquot before spectrophotometric measurement so that suspended ZnO does not distort absorbance.

Step 9: Determine removal and kinetics. Use a calibration curve to convert absorbance to dye

concentration. Calculate removal as $\text{Removal (\%)} = [(C_0 - C_t)/C_0] \times 100$. Evaluate apparent pseudo-first-order kinetics from $\ln(C_0/C_t) = k_{app} t$. Compare the slope, final removal and treatment rate across catalyst doses and pH values. The use of kinetic analysis is consistent with established ZnO photocatalysis studies [3,7].

Step 10: Evaluate recovery and reuse. After each run, recover ZnO by centrifugation, wash with water, dry at low temperature and reuse the catalyst for at least three cycles. Record the recovered mass and final removal efficiency. A progressive decline can indicate fouling, particle loss or surface deactivation. Recovery is essential because nanoparticle release is a practical environmental concern [2].

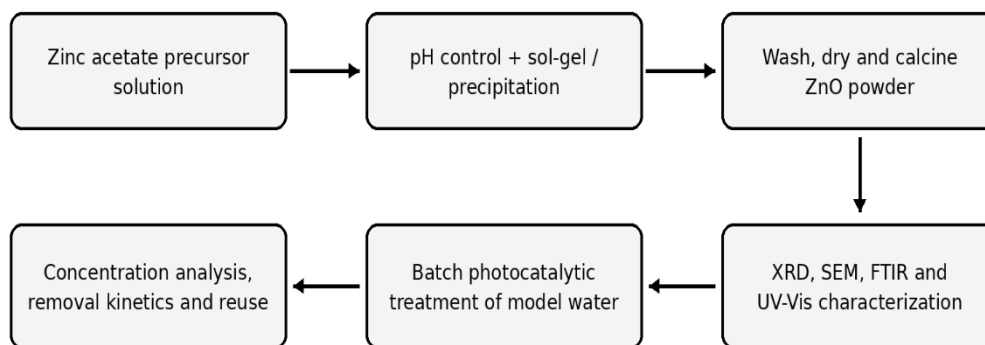


Figure 1. Proposed synthesis, characterization and wastewater-treatment workflow (original schematic).

Results

Because this is a methodology paper rather than a report of newly performed experiments, the results below are presented as validation targets derived from pre-2019 literature. They indicate the ranges that a successful laboratory implementation could reasonably be expected to approach, but they must not be reported as original measurements until the procedure is actually carried out.

Expected Characterization Outcomes

A successful ZnO synthesis should produce XRD reflections corresponding to crystalline wurtzite ZnO with no major precursor peaks. Balcha et al. [3] reported crystallite sizes of about 30 nm for precipitation-derived ZnO and 28 nm for sol-gel ZnO. SEM would be expected to show nanoscale primary particles together with some degree of agglomeration, which is common for high-surface-energy nanopowders. FTIR should show a low-wavenumber Zn-O vibration together with weaker bands associated with surface hydroxyl

groups. UV-Visible characterization should show strong near-UV absorption consistent with the wide band gap of ZnO. These measurements provide complementary confirmation of phase, morphology, surface chemistry and optical response rather than relying on a single instrument.

Wastewater-Treatment Performance Targets

For the photocatalytic test, concentration should decrease more rapidly in the illuminated ZnO suspension than in the photolysis blank or dark adsorption control. An optimum catalyst dose is expected: too little catalyst provides insufficient active area, whereas excessive loading can scatter light and reduce photon penetration. Balcha et al. [3] reported an optimum ZnO loading of 250 mg/L in their test range and higher methylene-blue degradation for sol-gel ZnO than for precipitation-derived ZnO. The broader ZnO literature similarly shows strong sensitivity to catalyst loading, pollutant concentration and pH [7].

Table 3. Literature-supported performance benchmarks

Material	Reported feature	Pollutant / target	Reported performance	Source
ZnO, precipitation	~30 nm crystallite	Methylene blue	~81% degradation; k about $8.4 \times 10^{-3} \text{ min}^{-1}$	[3]
ZnO, sol-gel	~28 nm crystallite	Methylene blue	~92.5% degradation; k about $12.4 \times 10^{-3} \text{ min}^{-1}$	[3]
Iron oxide	94.1 m ² /g surface area	As(V), Cr(VI), Congo red	39.6, 35.0 and 109.2 mg/g adsorption capacities	[6]
1% V-TiO ₂	Sunlight-active photocatalyst	Jakofix red / spent wash	~90% dye in 3.5 h; ~65% spent wash in 5 h	[9]

The benchmark data show why synthesis and characterization must be linked to treatment testing. Similar metal oxides can exhibit different removal rates when crystal size, surface defects, light absorption or morphology change. The iron-oxide example [6] also demonstrates that adsorption capacity and easy magnetic separation can be as important as photocatalytic rate, depending on the target contaminant.

Conclusion and Discussion

The proposed methodology provides a practical sequence for synthesizing and evaluating metal oxide nanoparticles for wastewater-treatment applications, using ZnO as the principal laboratory model. The procedure connects wet-chemical preparation, calcination, structural and optical characterization, controlled batch treatment, kinetic analysis and catalyst reuse. This connection is important because treatment performance cannot be interpreted confidently without knowing whether the intended oxide phase, crystallite scale and optical response were actually obtained.

The literature through 2018 supports the choice of wet-chemical synthesis and photocatalytic testing. Nikam et al. [1] showed that wet-chemical conditions can be tuned to control metal oxide nanoparticle properties. Lu et al. [2] placed ZnO, TiO₂ and iron oxides among the major nanomaterials investigated for water treatment, while Balcha et al. [3] demonstrated that changing the ZnO synthesis method alters both crystallite size and methylene-blue degradation. The broader ZnO review by Lam et al. [7] confirms that operating conditions strongly affect treatment efficiency. For TiO₂, Mahlambi et al. [4] and Reddy et al. [8] emphasized the importance of photocatalyst design and irradiation, and later pre-2019 studies demonstrated both greener synthesis [10] and sunlight-active treatment of complex effluents [9].

The method also has limitations that should be addressed in future experiments. First, methylene blue is a convenient model compound but does not reproduce the complexity of real industrial wastewater. After optimization with the model solution, the same catalyst should therefore be tested with filtered and unfiltered real effluent, with chemical oxygen demand, total organic carbon and toxicity included as response variables. Second, nanoparticle recovery is critical. Suspended ZnO that remains in treated water can create secondary contamination, so immobilization on a support, membrane-assisted recovery or magnetic composite design may be required at larger scale. Third, UV-dependent photocatalysis consumes energy and may not use the solar spectrum efficiently. Doping and composite formation can improve visible-light activity, but any modification must be evaluated for stability and leaching.

A further consideration is that maximum decolorization does not necessarily mean complete mineralization. Dye chromophores can be destroyed while organic intermediates remain. Accordingly, future work should pair UV-Visible decolorization with total organic carbon or chemical oxygen demand measurements. Catalyst stability should also be checked by XRD or FTIR after repeated cycles. These additions would strengthen the transition from a laboratory methodology to a credible wastewater-treatment process.

In conclusion, ZnO provides a suitable representative metal oxide for a 2018-cutoff methodology paper because it can be synthesized with simple reagents, characterized using standard instruments and tested quantitatively through photocatalytic degradation. Comparison with TiO₂ and magnetic iron oxides demonstrates that the broader metal-oxide platform supports multiple treatment mechanisms, including advanced oxidation and adsorption. The step-by-step protocol, controls and analysis equations presented here can be directly used as the basis

for experimental validation while keeping reported outcomes separate from literature benchmarks.

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