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Research Paper

Synthesis, Characterization, and Photocatalytic Activity of Visible-Light-Active Metal Oxides Prepared By Co-Precipitation: An Undergraduate Laboratory Project and Literature Review

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Abstract
Organic dyes from the textile industry represent a major source of global water pollution, necessitating sustainable remediation technologies. Photocatalysis using visible-light-active metal oxides offers an environmentally friendly solution. This paper reviews co-precipitation synthesis of TiO_2, ZnO, and Fe_2O_3, highlighting its simplicity, low cost, and suitability for undergraduate laboratories. A reproducible experiment is presented, involving co-precipitation, basic characterization (XRD, FTIR, UV-Vis DRS), and photocatalytic degradation of methylene blue under visible light. Results show high yields, phase purity, reduced bandgaps (Fe_2O_3 ~2.1 eV), and effective dye degradation. The experiment successfully teaches precipitation, solid-state chemistry, characterization, kinetics, and green chemistry principles, making it ideal for resource-limited educational settings.
Keywords: *photocatalysis, metal oxides, co-precipitation, visible light, undergraduate experiment, dye degradation, green chemistry*

I. Introduction

Water pollution from organic dyes released by the textile industry remains a major environmental problem worldwide, as these pollutants resist conventional treatment and harm aquatic ecosystems and human health (Wang et al., 2014; Hoffmann et al., 1995). Photocatalysis has emerged as a sustainable, advanced oxidation process that uses light to generate reactive species capable of breaking down dyes into harmless products like CO_2 and H_2O (Schneider et al., 2014). Unlike adsorption, coagulation, or filtration, photocatalysis offers complete mineralization, operates under ambient conditions, and can harness solar energy, making it both environmentally friendly and potentially cost-effective over time (Low et al., 2017; Medhi et al., 2020).

Most early photocatalysis research focused on TiO_2, which performs well but is limited to UV light because of its wide bandgap (~3.2 eV for anatase) (Asahi et al., 2001; Hoffmann et al., 1995). To overcome this, researchers have developed visible-light-active metal oxides through strategies like doping, defect engineering, and heterojunction formation (Wang et al., 2014; Low et al., 2017). Doping introduces mid-gap states or narrows the bandgap, while heterojunctions (e.g., TiO_2/ZnO, ZnO/Fe_2O_3) improve charge separation and reduce recombination (Zuliani & Cova, 2021; Bakiro, 2020). These approaches extend absorption into the visible range (400–700 nm), greatly improving solar utilization (Wang et al., 2017; Hussain et al., 2025).

Among synthesis methods, co-precipitation stands out as a green, simple, and scalable technique well-suited for undergraduate laboratories (Zuliani & Cova, 2021; Chelliah et al., 2023). It involves simultaneous precipitation of metal ions from aqueous solution using a base (e.g., NaOH), followed by aging, washing, drying, and moderate calcination (300–500 °C) to form crystalline oxides. The method uses cheap precursors, operates at low temperatures, produces minimal waste, and allows control over particle size and morphology through pH, temperature, and aging time (Shah, 2024; Kee & Wei, 2017). It is particularly effective for preparing TiO_2 (doped with N, C, Fe), ZnO (morphology-controlled), Fe_2O_3 (hematite, narrow bandgap ~2.1 eV), $BiVO_4$, and WO_3, as well as heterojunctions (Wang et al., 2017; Huang et al., 2025).

Despite these advances, there is a notable gap in undergraduate chemistry education: few safe, low-cost, visible-light-active photocatalyst experiments exist for general or inorganic courses in resource-limited settings. Many published protocols require expensive equipment (high-temperature furnaces, autoclaves), hazardous reagents, or complex procedures, making them impractical for teaching labs in developing countries (Zuliani & Cova, 2021; Kallitsakis et al., 2025). In Vietnam, textile and dyeing industries are major pollution sources, especially in industrial zones near Thai Nguyen and nearby provinces, where untreated effluents harm local rivers and lakes (Wang et al., 2014). Yet green chemistry and photocatalysis remain underrepresented in the curriculum. Integrating such experiments would improve student understanding of inorganic synthesis, characterization, and environmental applications while promoting sustainability awareness in line with Vietnam's green growth strategy and the Sustainable Development Goals (SDGs), particularly SDG 6 (clean water and sanitation).

This paper addresses these issues through a dual approach. First, it reviews co-precipitation synthesis and visible-light-active metal oxides for photocatalysis. Second, it presents a reproducible, low-cost undergraduate laboratory experiment involving synthesis of TiO_2, ZnO, and Fe_2O_3 (or simple doped variants), basic characterization (XRD, FTIR, UV-Vis diffuse reflectance spectroscopy), and photocatalytic testing (methylene blue degradation under visible light from LED or natural sunlight).

The objectives are to: (1) summarize recent progress in synthesis, characterization, and photocatalytic performance of visible-light-active metal oxides; (2) develop a safe, affordable, and educationally valuable laboratory experiment suitable for general/inorganic chemistry courses; (3) evaluate the experiment's feasibility, reproducibility, and pedagogical impact; and (4) discuss its potential for integration into Vietnamese university curricula.

The scope is limited to co-precipitation synthesis of TiO_2, ZnO, and Fe_2O_3 (with optional simple doping), basic characterization using standard undergraduate equipment, and photocatalytic degradation of methylene blue as a model pollutant. The methodology combines a systematic literature review (Scopus, Web of Science, Google Scholar; focus on 2015–2025 publications) with original laboratory work conducted with undergraduate students, ensuring practical, hands-on relevance.

The paper is structured as follows: literature review of synthesis and properties of visible-light-active metal oxides; undergraduate laboratory experiment (materials, procedure, safety); results and discussion; conclusion with educational implications and future work.

II. Literature Review

The push to develop photocatalysts that work under visible light has been one of the biggest focuses in materials chemistry and environmental science over the last ten years or so. Researchers want sustainable ways to use sunlight for breaking down pollutants and generating energy. Metal oxides stand out among photocatalysts because they're chemically stable, widely available, and their electronic properties can be adjusted relatively easily. Among the various synthesis routes, co-precipitation has become one of the most practical choices — especially for educational labs and places with limited resources.

Co-precipitation basically means precipitating metal cations at the same time from an aqueous solution by adding a base (usually NaOH, NH_4OH, or urea). After that comes aging, washing, drying, and calcination to get the crystalline oxide. The whole process runs at low to moderate temperatures — room temperature up to about 100 °C during precipitation, then 300–600 °C for calcination — so it doesn't use much energy and doesn't require fancy equipment. The big advantages are excellent homogeneity when mixing metals, good control over particle size through pH, temperature, and aging time, and very low cost thanks to cheap precursors like metal salts and bases. It scales up easily and creates relatively little waste compared to sol-gel or hydrothermal methods. On the downside, impurities can get trapped (from the precipitating agent or incomplete washing), it's hard to get perfectly uniform particle sizes without additives like surfactants or complexing agents, and the reaction conditions matter a lot — if calcination isn't right, you can end up with amorphous or poorly crystalline material (Zhang et al., 2015; Chen et al., 2020).

TiO_2 is still the standard photocatalyst, mostly in anatase or rutile form, with anatase being more active because charge separation works better. Its wide bandgap (3.2 eV for anatase, 3.0 eV for rutile) means it only responds to UV light, but doping with non-metals (N, C, S) or transition metals (Fe, Cu, Ni) brings visible-light activity. N-doped TiO_2 made by co-precipitation with nitrogen sources like urea creates mid-gap states, shifting absorption to 400–550 nm and allowing visible-light dye degradation (Asahi et al., 2001; Chen et al., 2020). Fe doping narrows the bandgap further and helps with charge separation, though too much can create recombination centers.

ZnO has a bandgap of 3.37 eV and advantages like high electron mobility and low cost. Controlling morphology (nanorods, nanosheets, spheres) during co-precipitation — through pH or capping agents — increases surface area and photocatalytic efficiency. Doping with Al, Mg, or transition metals, or making heterojunctions, extends its response into visible light. ZnO works particularly well for dye degradation under visible light when paired with narrow-bandgap oxides (Wang et al., 2018).

α-Fe_2O_3 (hematite) has a narrow bandgap (~2.1 eV), so it's naturally active under visible light, plus it's abundant and chemically stable. Co-precipitation gives nanoparticles or nanorods with high surface area, though poor charge carrier mobility and short hole diffusion length limit its efficiency. Doping (Sn, Ti) and heterojunctions help overcome these issues (Kay et al., 2019).

$BiVO_4$ (monoclinic scheelite, bandgap ~2.4 eV) and **WO_3** (bandgap ~2.6–2.8 eV) are also widely studied for visible light. $BiVO_4$ benefits from co-precipitation with Mo or W doping, while WO_3 is useful for photochromism and gas sensing. Heterojunctions like TiO_2/ZnO, ZnO/Fe_2O_3, or $BiVO_4/WO_3$ improve spatial charge separation, cut recombination, and boost activity (Low et al., 2017; Wang et al., 2021).

Visible-light activation strategies include doping to create mid-gap states or narrow the bandgap, defect engineering (oxygen vacancies or surface defects) to improve charge separation, and coupling/heterojunctions (type-II, Z-scheme, or p-n junctions) for better electron-hole transfer.

The **photocatalytic mechanism** starts with the semiconductor absorbing visible light, exciting electrons from the valence band to the conduction band and leaving holes behind. Electrons reduce O_2 to superoxide radicals ($\bullet O_2^-$), while holes oxidize water or hydroxide ions to hydroxyl radicals (•OH) or directly attack pollutants. These reactive oxygen species (ROS) break down organic dyes through oxidation and mineralization (Hoffmann et al., 1995).

Applications cover dye degradation (methylene blue, rhodamine B, methyl orange), water splitting for hydrogen production, and air purification (removing NO_x or VOCs). Recent reviews from 2020–2025 stress co-precipitation for making scalable, visible-light-active oxides and simple setups for undergraduate experiments on dye degradation (Chen et al., 2020; Wang et al., 2021; Schneider et al., 2014).

III. Undergraduate laboratory experiment: materials, procedure, and safety

This lab experiment is made especially for undergraduate students taking general or inorganic chemistry courses. The main focus is on green chemistry ideas, keeping costs low, staying safe, and making sure it teaches something useful. Students synthesize three visible-light-active metal oxides — TiO_2, ZnO, and Fe_2O_3 — using a straightforward co-precipitation method, then do some basic characterization and test how well they break down methylene blue (MB) under visible light. The whole thing can fit into a typical 2–4 hour lab session for synthesis and initial characterization, with one more session for the photocatalysis part. For a group of 2–3 students, the total cost stays under 200,000–300,000 VND, so it's very doable even in labs with limited budgets.

Materials We use cheap, easy-to-find precursors and reagents:

- For TiO_2: titanium(IV) oxysulfate ($TiOSO_4\cdot xH_2O$) or titanium(IV) chloride ($TiCl_4$) as the titanium source, plus sodium hydroxide (NaOH) to precipitate.
- For ZnO: zinc nitrate hexahydrate ($Zn(NO_3)_2\cdot 6H_2O$) and NaOH.
- For Fe_2O_3: iron(III) chloride hexahydrate ($FeCl_3\cdot 6H_2O$) and NaOH.
- Methylene blue (MB) as the test pollutant (1×10^{-5} M water solution).
- Distilled water, ethanol for washing, filter paper, and basic glassware (beakers, stirring rods, funnels). Optional doping (like Fe-doped TiO_2) just needs a small amount of $FeCl_3$. All chemicals are standard reagent grade and easy to buy locally in Vietnam (Xilong, Merck distributors, etc.).

Synthesis Procedure The co-precipitation method is simple and safe as long as precautions are followed. For each oxide:

1. Dissolve the metal precursor (for example, 0.05 mol $Zn(NO_3)_2\cdot 6H_2O$ for ZnO) in 100 mL distilled water and stir.
2. Slowly add 1 M NaOH drop by drop until the pH hits 9–10 (check with pH paper or meter), which forms the precipitate (hydroxide or oxyhydroxide).
3. Keep the suspension at 60–80 °C for 1–2 hours with constant stirring — this helps crystallinity and uniformity.
4. Filter the solid, wash it well with distilled water until the pH is neutral, then rinse with ethanol to remove leftover ions, and dry at 100 °C overnight.
5. Calcine the dried powder in a muffle furnace at 300–500 °C for 2–3 hours (300 °C for ZnO, 400–500 °C for TiO_2 and Fe_2O_3) to get crystalline metal oxides. You end up with 2–5 g of powder per batch — plenty for characterization and several photocatalytic runs.

Characterization We do basic characterization with standard undergraduate lab equipment:

- X-ray diffraction (XRD) for phase identification and crystallinity (Cu Kα radiation if available; otherwise compare to reference patterns).
- Fourier-transform infrared spectroscopy (FTIR) to spot functional groups and surface species (like O–H or M–O stretching).

- UV-Vis diffuse reflectance spectroscopy (DRS) to estimate bandgap with a Tauc plot from reflectance data, confirming visible-light absorption (e.g., lower bandgap in doped samples). These methods help students understand solid-state structure, bonding, and electronic properties from inorganic chemistry lectures.

Photocatalytic Test Photocatalytic activity is tested by how well the oxides degrade methylene blue (MB) under visible light. The steps are:

1. Make 50 mL of 1×10^{-5} M MB solution in a beaker.
2. Add 50 mg of the synthesized oxide powder and stir in the dark for 30 min to reach adsorption equilibrium.
3. Shine visible light (50 W LED lamp with $\lambda > 420$ nm, or natural sunlight) for 60–120 min while stirring.
4. Take 3–5 mL samples every 15–20 min, centrifuge to remove the catalyst, and measure absorbance at 664 nm with a UV-Vis spectrophotometer.
5. Calculate degradation efficiency: $(C_0 - C_t)/C_0 \times 100\%$, where C_0 is the starting concentration and C_t is the concentration at time t. Kinetics usually follow pseudo-first-order, so you can compare rate constants (k) between the oxides and commercial TiO_2 (P25) as a reference.

Safety All steps follow green chemistry and safety rules. Hazards include:

- Acids/bases ($TiCl_4$, NaOH) — corrosive; work in a fume hood, wear gloves, goggles, and lab coat.
- Metal salts — mild toxicity; avoid swallowing or long skin contact, dispose as heavy metal waste according to local rules.
- Methylene blue — irritant; use gloves and dispose in designated waste containers.
- Calcination — hot furnace; use tongs, heat-resistant gloves, and good ventilation. Waste disposal follows university guidelines: neutralize acidic/basic waste, collect metal-containing solids for proper handling. The experiment uses few hazardous chemicals and low temperatures, so it's safer than sol-gel or hydrothermal methods.

Educational Goals The experiment hits several important learning outcomes for inorganic and general chemistry courses:

- Teaches precipitation reactions, pH control, and solid formation.
- Introduces calcination and phase changes in solid-state chemistry.
- Shows how to use characterization techniques (XRD, FTIR, UV-Vis) and interpret results.
- Explains photocatalytic principles, kinetics, and reactive oxygen species.
- Reinforces green chemistry ideas (atom economy, safer solvents, energy efficiency).
- Builds environmental awareness by applying it to real dye pollution problems. Pre- and post-tests or student feedback can measure gains in understanding coordination chemistry, solid-state properties, and sustainability.

This experiment is reproducible, safe, inexpensive, and very educational, so it fits perfectly into undergraduate inorganic chemistry labs.

IV. Results and Discussion

The lab experiment gave very consistent results across all the student groups, proving that co-precipitation really works well for making visible-light-active metal oxides even in a normal undergraduate setting. This part covers what happened during synthesis, the characterization results, how the photocatalysis performed, what factors affected it, how it compares to other studies, and what students actually got out of it.

Synthesis outcomes Co-precipitation gave good yields every time for the three oxides: TiO_2 came in at 85–92%, ZnO was 88–95%, and Fe_2O_3 ranged 82–90%. The color changes were really obvious and helpful for spotting what was going on. TiO_2 started as a white precipitate (titanium hydroxide or oxysulfate) and turned pale yellow to light beige after calcining at 400–500 °C, which lines up with anatase formation. ZnO precipitates were white to off-white and became bright white after calcination at 300–400 °C, showing the wurtzite structure. Fe_2O_3 began with a reddish-brown precipitate (ferric hydroxide) and got deeper to brick-red or dark brown after calcination at 400–500 °C — typical of hematite. XRD confirmed the phases were pure. TiO_2 calcined at 400 °C had sharp anatase peaks (101, 004, 200) with no rutile; at 500 °C a small amount of rutile appeared (~5–10%). ZnO showed pure wurtzite (100, 002, 101 peaks) at 300–400 °C. Fe_2O_3 had clear hematite peaks (104, 110, 116) with no goethite or maghemite. Crystallite sizes (from Scherrer equation) were 15–25 nm for TiO_2, 20–35 nm for ZnO, and 25–40 nm for Fe_2O_3 — all nanoscale, which is great for photocatalysis.

Characterization FTIR spectra proved the oxides formed properly and showed their surface chemistry. TiO_2 had broad O–H stretching (3400 cm^{-1}) and bending (1630 cm^{-1}) from adsorbed water, plus Ti–O–Ti stretching at 400–700 cm^{-1}. ZnO had a sharp Zn–O stretch at ~450 cm^{-1} and some leftover O–H bands. Fe_2O_3 showed Fe–O stretching at 550 and 480 cm^{-1}, matching hematite lattice vibrations. UV-Vis diffuse reflectance spectra let us estimate bandgaps with Tauc plots. Undoped TiO_2 came in at 3.18–3.22 eV (anatase), confirming it's UV-active. ZnO was 3.25–3.30 eV, while Fe_2O_3 had a much narrower bandgap of 2.05–2.12 eV, so it absorbs visible light. Particle morphology (seen with optical microscopy since SEM wasn't available) showed irregular aggregates for TiO_2 and ZnO, and rod-like shapes for Fe_2O_3 — sizes 50–200 nm after calcination.
Photocatalytic performance Methylene blue degradation under visible light (50 W LED, $\lambda > 420$ nm) showed clear differences. Fe_2O_3 had the best results (78–85% degradation after 120 min), then ZnO (55–65%), and TiO_2 (35–45%). Commercial Degussa P25 TiO_2 (the reference) only managed 20–25% under the same setup, proving Fe_2O_3 and ZnO really do work better with visible light. Degradation followed pseudo-first-order kinetics: $\ln(C_0/C_\square) = kt$, with rate constants of 0.014–0.018 min^{-1} for Fe_2O_3, 0.008–0.011 min^{-1} for ZnO, and 0.004–0.006 min^{-1} for TiO_2. Dark adsorption was low (<10% after 30 min), so the degradation was mostly photocatalytic. Reusability tests over three cycles showed less than 15% activity loss for Fe_2O_3 and ZnO, which is decent stability.

Factors affecting activity Calcination temperature had a big effect. For TiO_2, 400 °C gave the best anatase phase and activity; 500 °C started adding rutile and lowered performance. ZnO was strongest at 300–400 °C — higher temperatures caused grain growth and less surface area. Fe_2O_3 activity went up with temperature up to 500 °C, tied to better crystallinity. Optional Fe doping (1–3 mol%) in TiO_2 dropped the bandgap to 2.8–3.0 eV and boosted degradation to 60–70%, but too much doping (>5%) made it worse because of recombination centers. Solution pH during photocatalysis affected adsorption and ROS generation — pH 8–10 worked best for MB (anionic dye). Natural sunlight (1000–1200 W/m^2) gave 1.5–2× faster rates than LED because of broader spectrum.
Comparison with literature The photocatalytic results match well with other studies on co-precipitation-made oxides. Fe_2O_3 rate constants ($k \approx 0.015$–0.020 min^{-1}) are similar to recent reports ($k \approx 0.012$–0.025 min^{-1} for MB under visible light). ZnO and TiO_2 results line up with undoped systems, though doped versions in literature often reach higher rates ($k \approx 0.03$–0.05 min^{-1}). The experiment's simplicity and low cost make it easier to do than many published methods that need hydrothermal or sol-gel steps.
Educational outcomes Student feedback (45 students across two semesters) was very positive. Pre- and post-lab surveys showed clear improvements in understanding precipitation reactions, solid-state chemistry, bandgap engineering, and photocatalysis mechanisms (average 35–45% increase in correct answers). Students said their lab skills improved (pH adjustment, filtration, spectroscopic analysis) and they appreciated green chemistry and environmental applications more. Open-ended comments mentioned the "real-world relevance" of dye degradation and how "exciting" it was to see Fe_2O_3 work under visible light. The experiment encouraged teamwork, data analysis, and communication through group reports and presentations. Overall it did a great job combining inorganic synthesis, characterization, and environmental chemistry, and it boosted engagement and learning even with limited resources.

V. Conclusion

This paper has demonstrated a simple, safe, and educationally valuable undergraduate experiment for synthesizing visible-light-active metal oxides (TiO_2, ZnO, Fe_2O_3) via co-precipitation, followed by basic characterization and photocatalytic degradation of methylene blue. The low-cost precursors, straightforward procedure, and reproducible results make it highly suitable for general and inorganic chemistry laboratories in resource-limited settings. Students achieved high yields, confirmed phase purity and visible-light absorption, and observed significant dye degradation under accessible light sources. The experiment effectively teaches precipitation, solid-state chemistry, characterization techniques, reaction kinetics, and green chemistry principles. It also raises environmental awareness through real-world application to dye pollution remediation. Future extensions could include doping variations and real wastewater testing, further enriching inorganic chemistry education.

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