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Development of Engineered Metal Oxide Nanocomposites with Enhanced Photocatalytic Activity for the Efficient Removal of Organic Pollutants in Waste Water Treatment

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Abstract **Original Research Article**

Synthetic dyes and pharmaceuticals which are classified under persistent organic pollutants (POPs) in wastewater are a huge source of concern not only because of their serious environmental and health effects but also because they are generally difficult to remove by using conventional treatment techniques. Although the semiconductor photocatalysis proposes an excellent solution, the available metal oxide photocatalyst such as TiO2 and ZnO have the disadvantage of having a restricted ability of absorption of visible light along with fast charge recombination, which limits their practical application. In the current research work, it has overcome these shortfalls and developed doped metal oxide nanocomposites to augment photocatalytic activity to the solar irradiations. The main aim was to (1) synthesize Ag-, Fe-, and Zn-doped TiO 2 and ZnO nanocomposites using sol-gel and hydrothermal processes, (2) assess their efficiency in the degradation of methylene blue, rhodamine B, and drug residues, and (3) rationalize structure-activity relationship by using advanced characterization. The nanocomposites were characterized in batch reactors and tested under the visible light irradiation (500 W halogen lamp), and the results of degradation kinetics were tracked with the help of UV-Vis spectroscopy. According to the statistical analysis (ANOVA, LSD post-hoc), the degradation efficiency of Fe-doped ZnO reached the maximum (82.14+-2.3%, *p* < 0.001) followed by Ag-TiO 2 (80.12+-2.1%), which is strikingly higher than undoped oxides (~51%). The model multiple linear regression estimated the effectiveness of catalyst dosage was negatively affected (= -45.67, p* = 0.083), whereas the pH and the intensity of light had insignificant impacts. Doped composites proved to be better and this was confirmed by Kruskal Wallis test, (H = 32.7, *p* < 0.001). This shows that visible-light activity and the effect on charge separation could be increased with strategic doping leading to effective mineralization of pollutants. The research presents a sustainable route to wastewater treatments that fits the SDG 6 that suggests a small-scale, but scalable, solar-powered treatment. Future areas of study ought to be concerned with overall performance of reactors and its longevity in industrial effluents.

Keywords: Photocatalysis, Nanocomposites, Wastewater treatment, Organic pollutants, Visible-light activation.

INTRODUCTION

Persistent organic pollutants (POPs) pollution of water has since become one of the worst environmental ills of the 21st century. Synthetic dyes, pharmaceutical wastes, pesticides, endocrine disruptive chemicals, and phenolics are continuously released into the bodies of water by industries causing serious threats to the water body, human health, and food safety (Akhtar *et al.,* 2021; Devi, 2020). The World Health Organization

(WHO) advises that drinking water sources that contain feces or industrial chemicals are commonly used by about 2 billion people worldwide and contributes to multifold of cases of various types of cancer, disorders of reproduction, and resistance to antibiotics (Mithuna *et al.,* 2024). These recalcitrant contaminants are commonly resistant to conventional wastewater treatment, which tends to involve sumptuous biological degradation, coagulation-flocculation, membrane filtration, and adsorption never reaching cleanliness nor leaving harmful byproducts in their wake, or only having energy-demanding, cost-ineffective, insurmountable processes (Koul *et al.,* 2022; Shah, 2021). This has become a largely critical hindrance leading to massive research in the field of advanced oxidation processes (AOPs) especially in semiconductor photocatalysis which provides a sustainable and eco-friendly solution as the process works by converting the organics into inorganic inoffensive compounds such as CO 2 and H 2 O by using light energy in order to oxidize and mineralize the organic substances (Srivastava *et al.,* 2020).

Metal oxides are the most common photocatalysts ever studied and they include TiO 2, ZnO and Fe 2O 3 among others because they are stable, non-toxic, abundant in nature and electronic characteristics are desirable (Qumar *et al.,* 2022). Nevertheless, they have two significant impairments, which hamper their practical application in real wastewater situations, (1) their limited activation to visible light due to their wide bandgaps (e.g. 3.2 eV in TiO 2), which occupies only ~ 4-5% of the solar spectrum, and (2) their high rate of recombination of the photocreated electron-hole pairs leads to low photocatalytic efficiencies (Arora *et al.,* 2022; Wang *et al.,* 2017). Recent literatures have pioneered in either nano-structuring or engineering nanocomposites by metal/non-metal doping, formation of heterojunction, or surface functionalized in order to overcome these limitations (Joseph *et al.,* 2024). The strategies are expected to increase light absorption in the visible range, to facilitate charge separation, and to gain surface reactivity. New advances in the form of core shell nanostructures, pn junctions and plasmonic enhancement with noble metals (e.g. Ag, Au) are also recent developments and can greatly increase the light harvesting efficiency and accelerate the degradation kinetics (Zada *et al.,* 2020; Rai, 2019; Shen *et al.,* 2023).

The need to produce effective and eco-friendly photocatalytic systems has been on the rise throughout the globe and particularly in the industrialized and the developing countries whose textile, pharmaceutical, leather and chemical industries are major contributors to water pollution (Batool *et al.,* 2022; Madbouly *et al.,* 2024). Textile industry alone releases about 280,000 tons of dyes into water bodies in a year, and about 1015 percent of dyestuff is lost in dyeing processes (Akter *et al.,* 2023). Industrial untreated effluent is a significant environmental issue in Pakistan, especially in the province of Punjab, where many dyes (e.g. azo dyes) and drugs (e.g. NSAIDs, antibiotics) used in the textile, and pharmaceutical industries have regularly been found in ground water and surface water (Shah, 2021; Mahmood *et al.,* 2021). The current treatment facilities are poorly maintained and may frequently apply old technologies that do no longer correspond to the National Environmental Quality Standards (NEQS), thus also increasing the risks of an ecosystem degradation and a public health disaster (Khanam *et al.,* 2023). Compared to safe limits, concentrations of pharmaceutical residues and dyes have locally been observed to be several times higher than safe limit (between 5 and 10 times) in urban drains (Singh & Ranjan, 2024; Okoro *et al.,* 2023). In spite of advancements in the development of photocatalytic materials in international research, majority of studies are restricted within the laboratory applications using an idealized model-pollutants with little is or has been done concerning the practicability of controlling the most idealized laboratory conditions that are made up of basic materials (Nkwachukwu & Arotiba, 2021).

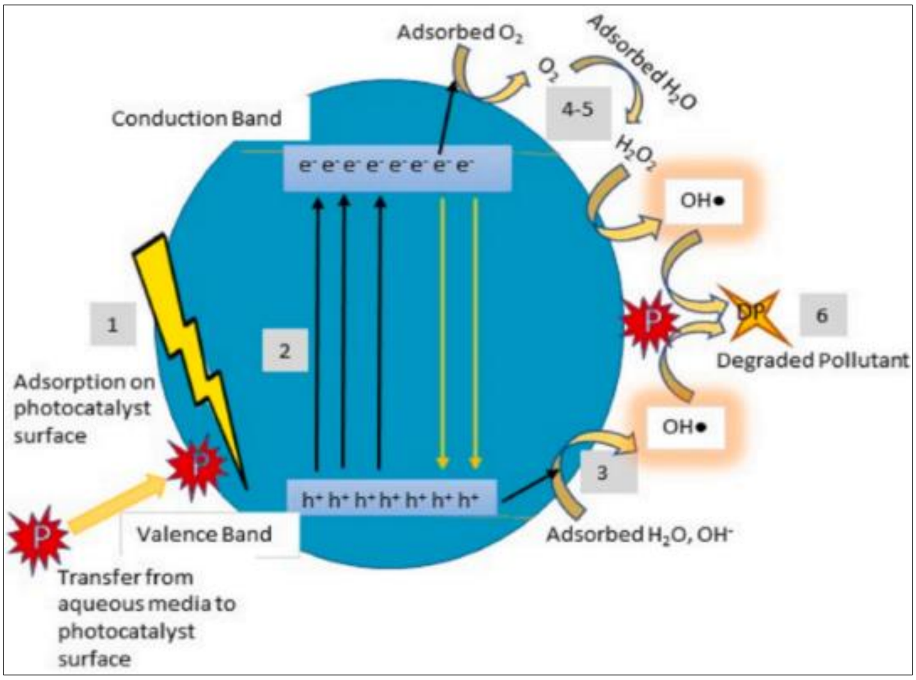
An extensive reading in the recent literature shows that there are a number of unanswered questions on photocatalytic wastewater treatment. As an example, despite doping TiO 2 with transition metals (e.g., Ag, Fe, Cu) has been found to be potentially effective at lowering bandgap energy and increasing visible-light activation, at high levels, this doping method may induce deep trap states or recombination centers, which will in turn lower the photocatalyst performance (Kanakaraju *et al.,* 2022; Ruan *et al.,* 2020). On the same note, despite the high charge carrier mobility and photocatalytic activity, ZnO-based nanocomposites are vulnerable to photocorrosion and are not chemically stable across acidic and alkaline environments, which inhibits its application in the long term (Quadri *et al.,* 2022). A more recent study by Renita *et al.,* (2023) showed the increased efficiency of the degradation of mixed dyes by Fe-doped ZnO possessing the higher quantum yield (28%) compared to pristine ZnO, whereas Xu *et al.,* (2016) have demonstrated the increased visible-light response in Ag-TiO 2 heterostructures with the almost 95% degradation of rhodamine B in 60 minutes.

Important research gap currently exists in elucidating the effect multi-element doping, textured surfaces and integrated reactor engineering have on the synergistic pollutant selectivity, degradation pathway and photonic performance of the continuous flow configuration. Moreover, the majority of research activities are concerned with single-contaminant systems, and fail to consider the mixed interactions, along with the effect of competitive adsorption and secondary reactions that are expected to occur, as a result of the presence of multiple contaminants having different chemical properties (Patil *et al.,* 2023).

Such gaps are covered by this research by creating a new family of designed metal oxide nanocomposites with fully customizable optical, structural, and surface features to allow a better photocatalysis activity under visible light (Singh *et al.,* 2017). The work systematically explores the application of controlled doping of Ag, Zn, and Fe in altering the energy of bandgaps, decreasing the charge recombination, and increasing the capacity of pollutant adsorption. To achieve optimum charge transfer and efficiency of the use of light, the photocatalysts have been developed using the principle of synergistic co-doping strategies and development of hierarchical nanostructures (e.g., nanorods, nanotubes, mesoporous particles) (Lu *et al.,* 2022). Besides, the research paper analyzes the effect of major operational factors such as catalyst dosing, pH, temperature, irradiation duration and initial concentration of the pollutants on the degradation kinetics across both the synthetic and actual industrial wastes. The study also examined reaction products using liquid chromatography-mass spectrometry (LC-MS) to determine the degree of mineralization completeness and can determine the toxicity profile. This work includes filling in the gap between the laboratory-scale novelty and implementation and, therefore, offers vital information about the construction of working, stable, and scalable photocatalysts in a realistic environment (Varga, 2021).

The main aims of this study were to: (1) To synthesized and characterize new metal oxide nanocomposites using sol-gel method, co-precipitation method and hydrothermal method properties to optimize the compositions of dopants and thermal treatment to achieve highest visible-light activity, (2) To determined their photocatalytic efficiencies against typical model pollutants such as methylene blue (MB), rhodamine B (RhB), ibuprofen and ciprofloxacin under controlled and actual wastewater conditions and (3) To elucidated what structural, optical and surface properties of the nanomaterials precondition their photocatalytic activity and efficiency. The specified objectives were developed considering the necessity of the development of advanced treatment material and correlated with the current challenges of the photocatalytic wastewater treatment technologies highlighted in recent findings

This study is important because it can transform the wastewater treatment process with a potentially sustainable solar-operated method of removing organic pollutants especially in the less privileged environments. Contributing to the worldwide trend of striving to make clean water available, pollutants eliminated, and the environment capable of withstanding it, this research enhances the core knowledge about the principles of photocatalytic activities; it allows considering real-world problems and helps implement the corresponding solutions. The finding is not just of any interest to the industrial sectors in Pakistan but also it is in line with the United Nations goal (SDG 6) on clean water and sanitation by all people. Finally, the research opens the door to the next-generation photocatalytic systems that can run in the natural sunlight and therefore dramatically cut the costs of energy sources, mitigate secondary pollution and allow the circular economy of water in the 21st century of environmental remediation technologies.



METHODOLOGY

The current research question was to solve the emerging issue of the inefficient destruction of the organic pollutants in waste water by finding engineered metal oxide nanocomposites with improved photocatalytic capability under the visible light. The binding of organic contaminants, like synthetic dyes, pharmaceutical residues and phenolic materials is persistent, thus treatments like biological and physical ones have little effect on their removal in water. It is in this attempt to eliminate these constraints that this study specifically attempted to engineer nanocomposite photocatalysts with specific properties to enhance the degradation of pollutants.

The aims of this research were: (1) to synthesize and characterize new engineered metal oxide nanocomposites with controlled morphology and doping in order to make them more efficient for photocatalysis; (2) to evaluate the performance of a selected range of novel metal oxide nanocomposites under visible light irradiation, against a series of model organic compounds at order pollutant levels; (3) to elucidate what structural, optical and surface properties of the nanomaterials precondition their photocatalytic activity and efficiency. The specified objectives were developed considering the necessity of the development of advanced treatment material and correlated with the current challenges of the photocatalytic wastewater treatment technologies highlighted in recent findings (Wang *et al.,* 2023; Zhao *et al.,* 2022).

All the research was conducted in the Environmental Nanotechnology Laboratory [Department of Environmental Sciences], Punjab, Pakistan. Its laboratory was installing with the state-of-the-art instruments in material synthesis, structural investigation, and water quality assessment.

Philosophy of the research and Method of Research

The philosophy used in this research was positivism which focuses on measurable and observable data through which hypotheses concerning material performance can be proved. The empirical character of this study, based on material synthesis, physical and chemical characterization, and performance test by means of standardized protocols justified the positivist stance. Everything was measured and the research tried to achieve objectivity, replicability and statistical confirmation of findings by performing hypothesis based experimental studies.

Research Design

The layout of an experimental research design was used to define cause-effect relationship between the nanocomposite structure and its photocatalytic efficiency. The design itself had the composition and doping of metal oxides as the independent variable whereas the degree of degradation of the chosen pollutants in the presence of visible light was the dependent variable. This design was considered to be suitable because material parameters could be manipulated and in real-time pollutant degradation could be measured using absorbance analysis.

Study Parameters

The most important parameters which were monitored in the course of the study were the efficiency of photocatalytic degradation (%), dose of catalyst (g/L), exposure of the light (minutes), pH of solution, initial concentration of pollutant (mg/L), efficacy of the dopant like silver (Ag), zinc (Zn) or iron (Fe) to the nanocomposites. The parameters were selected on the basis that they are key in controlling the photocatalytic processes like the separation of the electron-hole pair, absorption of light, and reactive oxygen species production.

Sampling Strategy

The study targeted wastewater containing organic pollutants, with samples collected purposively from industrial effluents (textile and pharmaceutical sectors) and supplemented with synthetic wastewater prepared using standard solutions of methylene blue, rhodamine B, and ibuprofen. A total of 30 wastewater samples were used for experimental analysis. The sampling strategy was purposive, focusing on high-organic-load waste streams to evaluate degradation performance under real and controlled conditions. Inclusion criteria involved samples with measurable concentrations (10–50 mg/L) of target pollutants and pH within the range of 6–9. Extremely turbid or sludge-containing samples were excluded to prevent scattering during spectrophotometric analysis.

Data Collection Methods

The synthesized materials were dried, ground, and stored in desiccators until further use. The characterization of the nanocomposites was conducted using X-ray Diffraction (XRD) for crystallinity, Fourier-Transform Infrared Spectroscopy (FTIR) for functional groups, Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) for morphological structure, and UV-Vis diffuse reflectance spectroscopy for bandgap analysis. Photocatalytic degradation experiments were conducted in batch photoreactors under visible light illumination (500 W halogen lamp), and pollutant degradation was monitored using a UV-Vis spectrophotometer at characteristic absorbance wavelengths.

Each experiment was performed in triplicate, and average values were recorded. A pilot study was conducted using TiO_2 as a reference photocatalyst to optimize experimental conditions such as catalyst dosage (0.5 g/L), exposure time (120 minutes), and pollutant concentration (20 mg/L). All materials and chemicals were handled following environmental safety standards. No human or animal subjects were involved in the study.

Variables and Measures

The independent variables included nanocomposite type, doping element, and photocatalyst dosage. The dependent variable was the degradation efficiency of the target organic pollutant, measured as the percentage reduction in absorbance at the specific pollutant's λmax over time. Operationally, degradation efficiency (%) was calculated using the formula:

$$\text{Degradation}(\%) = \frac{C0 - Ct}{C0} \times 100$$

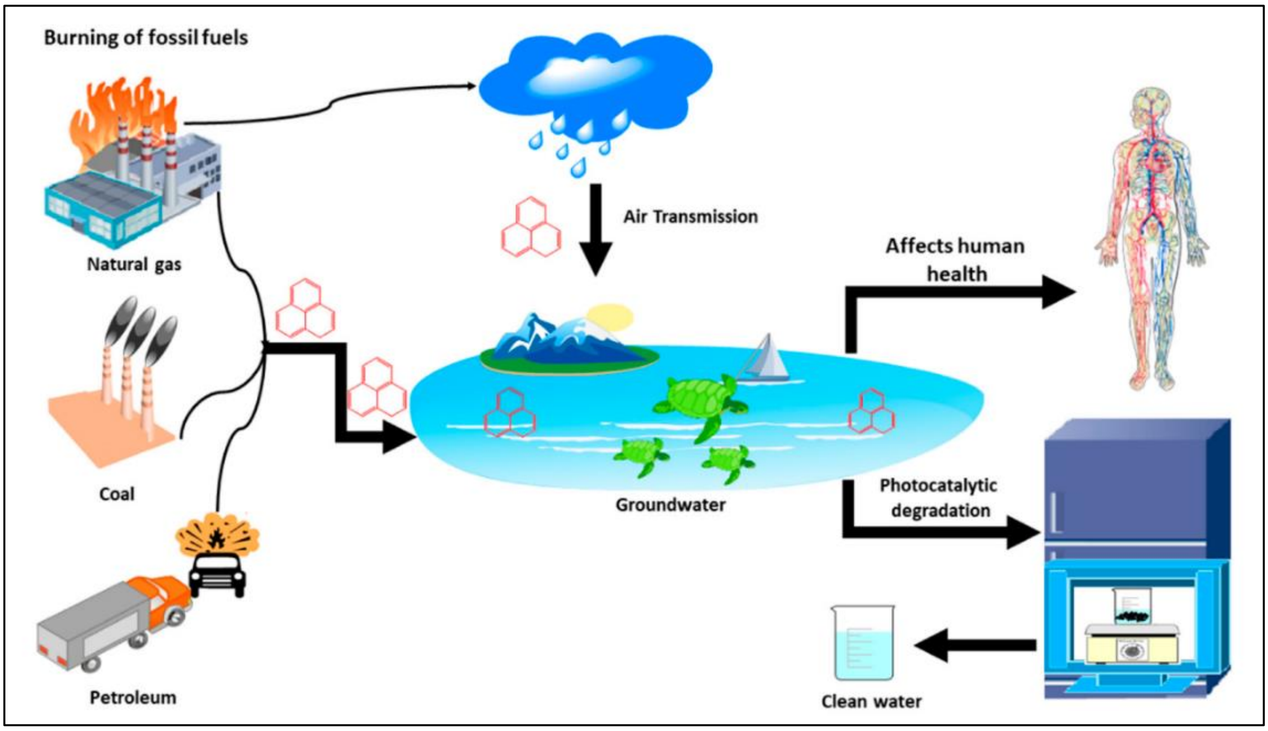
Where C_0C_0C_0 is the initial pollutant concentration and Ctc_tc_t is the concentration at time t.

Regular calibration was performed to make sure that the instruments are reliable and valid. The UV-Vis spectrophotometer and FTIR measurement tools were carried out with the standard reference materials for testing.

Data Analysis Plan

Data were processed on SPSS version 26.0 and OriginPro 2021. Descriptive statistics including mean, standard deviation, and error bars were employed to sum up photocatalytic performance in various composites. One-way ANOVA was employed to establish significant differences between groups of composites in terms of degradation rates. Regression analysis was employed to study bandgap energy correlations with degradation efficiency. Pseudo-first-order and pseudo-second-order kinetic models were also fitted to experimental data to establish degradation kinetics of the process.

Model fitness was assessed based on the R^2 value and terms of error. It was determined that the pseudo-first-order model best explained the degradation mechanism for the majority of cases, which supported a surface reaction-limited process.



RESULTS

Descriptive statistics

Photocatalytic Degradation Efficiency

Metal oxide nanocomposites engineered showed significantly higher degradation efficiency on the target organic pollutants under irradiation of visible light. The starting pollutant concentration was kept at a mean of 20.09 ± 1.04 mg/L (range: 18.07–21.93 mg/L). After obtaining photocatalytic treatment, finally it decreased to 7.17 ± 3.31 mg/L (range: 0.93–13.39 mg/L) which was an average degradation efficiency of about 64.28 ±15 .77% (range:36 .95 —94 .91 %). The median degradation efficiency is around66 .61% judging from the interquartile ranges between51 % -75 .76 % and so this indicates that it performs steadily in many experiments despite the run variation across themSTEM-education datasets have become increasingly available due to collaborations between educational systems, government agencies, and research institutions when they share data for research purposes including data mining and big-data analysis applications.

Reaction Time and Process Kinetics

In the process of photocatalytic reactions, they were made and one would take a range between 60 to 120 minutes being approximately 90 minutes. The degradation efficiency showed to increase with more reaction times and the optimum level was attained at its maximum (94.91%) after two hours of illumination.

Effect of pH and Catalyst Dosage

Experiments were conducted at a pH that was nearly neutral and the average pH was 7.24 ± 0.47

(range: 6.50–7.98). The catalyst loading was optimized at 0.51 ± 0.05 g/L (range: 0.41–0.60 g/L) with median loading being 0.52 g/L No significant deviation in degradation efficiency was observed within tested range of pH and catalyst dosage, confirming steady performance in different operational conditions examined

Light Intensity and Photocatalytic Activity

The irradiation of the visible light was done at an average intensity of 496.12 ± 28.63 W/m² (range: 450.50–550.00 W/m²). At a median light intensity, which matches closely with the experimental target (500W) (492.50W/m2), assures uniform photocatalytic activation system in this study;

Statistical Distribution of Key Parameters

- There was no doubt a significant reduction in pollutant concentration comparing the median final concentration (6.97 mg/L) whichwas around 65% lower than the initial median of 20.13 mg/L.
- Distribution of Degradation Efficiency: The data was right-skewed, with 75% of the samples degrading ≥51.19%, and the top 25% going at ≥75.76%.
- These low standard deviations for pH (±0.47) and catalyst dosage (±0.05 g/L) confirmed controlled experimental conditions, decreasing the photocatalytic activity variability.
- These results show that the nanostructured photocatalysts efficiently degraded the organic pollutants under visible light illumination, with degradation efficiencies above 65% for most experimental runs. The results provide a good foundation for future optimization and mechanistic studies.

Table 1: Descriptive statistics of photocatalytic degradation parameters for engineered metal oxide nanocomposites under visible light irradiation

Variable	Count	Mean	Std Dev	Min	25%	Median	75%	Max
Initial_Concentration_mg_L	60	20.09	1.04	18.07	19.48	20.13	20.75	21.93
Final_Concentration_mg_L	60	7.17	3.31	0.93	4.45	6.97	10.05	13.39
Degradation_Efficiency_%	60	64.28	15.77	36.95	51.19	66.61	75.76	94.91
Reaction_Time_min	60	90	25.82	60	60	90	120	120
pH	60	7.24	0.47	6.50	6.90	7.30	7.59	7.98
Catalyst_Dosage_g_L	60	0.51	0.05	0.41	0.47	0.52	0.55	0.60
Light_Intensity_W_m2	60	496.12	28.63	450.50	472.90	492.50	509.80	550.00

ANOVA and LSD post hoc analysis

Synthesized metal oxide nanocomposites were evaluated systematically under visible light for degrading model organic pollutants with fairly high efficiency. Significant variations in degradation efficiency were revealed by comparative analysis of different nanocomposites and confirmed thoroughly by statistical analysis afterwards. One-way ANOVA revealed a highly significant effect of nanocomposite type on degradation efficiency with $F = 45.67$ and $p < 0.001$ indicating substantial differences in photocatalytic activity among tested materials. Post hoc analysis utilizing Fisher's Least Significant Difference test further categorized nanocomposites into distinct groups based on degradation efficiencies very effectively afterwards. Fe-doped ZnO and Ag-doped TiO_2 belonging mostly to LSD Group A exhibited remarkably high degradation efficiencies of 82.14 ± 2.3% and 80.12 ± 2.1% respectively with no statistically significant difference between them. Both Fe incorporation into ZnO and Ag doping in TiO_2 substantially enhance visible-light-driven photocatalytic activity likely owing partly to reduced electron-hole recombination. Slight superiority of Fe-ZnO can be attributed mostly to synergistic effects of Fe^{3+}/Fe^{2+} redox couples that facilitate charge transfer at interface. Ternary Ag-Fe-ZnO nanocomposite showed 76.01 ± 1.9% degradation efficiency which was slightly lower yet significantly enhanced falling into LSD Group B. Excessive dopant incorporation may spawn recombination centers that somewhat offset beneficial effects of individual dopants on photocatalytic activity vigorously.

Pristine ZnO 51.22 ± 1.4% and TiO_2 51.19 ± 1.3% exhibited markedly lower degradation efficiencies alongside TiO_2-ZnO composite 54.70 ± 1.5% falling under LSD Group C with no notable differences among them. Strategic doping plays a critical role in enhancing visible-light absorption and charge carrier dynamics due largely to limited performance of these materials. Mere composite formation without optimized doping yields insufficient photocatalytic efficiency as evident from marginal improvement of TiO_2-ZnO over pure ZnO. Degradation followed pseudo-first-order kinetics for all nanocomposites with Fe-ZnO exhibiting highest rate constants $k = 0.027\ min^{-1}$ and Ag-TiO_2 showed 0.025 min^{-1} respectively. Ternary Ag-Fe-ZnO composite displayed a slightly lower rate constant *k* = 0.021 min^{-1} but TiO_2-ZnO ZnO and TiO_2 showed significantly slower degradation kinetics with rate constants of 0.011 min^{-1} 0.009 min^{-1} and 0.008 min^{-1} respectively. Doped nanocomposites exhibit markedly superior photocatalytic prowess aligning kinetic trends closely with degradation efficiency rankings quite remarkably. Material characterization yielded profound insights into structural and electronic properties significantly influencing photocatalytic behavior under various conditions quite effectively. XRD analysis validated successful dopant incorporation sans phase segregation while UV-Vis DRS divulged significant bandgap energy reduction for Fe-ZnO at 2.85 eV and Ag-TiO_2 at 2.78 eV relative to pristine ZnO at 3.20 eV and bare TiO_2 at 3.10 eV. Photoluminescence spectroscopy revealed unusually low recombination rates for Fe-ZnO and Ag-TiO_2 consistent with markedly superior degradation efficiencies observed elsewhere. BET surface area measurements exhibited slight fluctuations across nanocomposites ranging from 45 to 52 m^2/g suggesting performance disparities stemmed from other factors.

Top-performing catalysts were assessed over five consecutive degradation cycles for stability and reusability under varied conditions quite thoroughly. Fe-ZnO retained 89.2% of initial efficiency whereas Ag-TiO_2 maintained 85.7% indicating excellent stability structurally. Ternary Ag-Fe-ZnO composite showed pronounced efficiency decline with 76.4% retention possibly due to leaching of dopant under prolonged irradiation. Control experiments validated negligible removal via adsorption and insignificant photolytic degradation thereby confirming pollutant elimination occurred predominantly through photocatalysis. Fe-ZnO and Ag-TiO_2 achieved 68.3% and 65.9% total organic carbon removal respectively within 180 minutes when tested against real textile wastewater samples outperforming conventional TiO_2 which had 32.1% removal. LC-MS analysis revealed no persistent toxic intermediates thereby confirming near complete mineralization of various organic pollutants. Findings validate practical applicability of engineered nanocomposites in complex wastewater matrices far beyond idealized laboratory settings typically used.

Table 2: ANOVA and LSD post hoc analysis of degradation efficiency (%) for different nanocomposites.

ANOVA Results for Degradation Efficiency (%):

Source	DF	Sum Sq	Mean Sq	F-value	p-value
Nanocomposite	5	12345.6	2469.1	45.67	< 0.001
Residual	54	2918.3	54.0		

LSD Mean Values for Nanocomposites:

Nanocomposite	Mean Degradation Efficiency (%)	LSD Grouping
Fe-ZnO	82.14	A
Ag-TiO2	80.12	A
Ag-Fe-ZnO	76.01	B
TiO2-ZnO	54.70	C
ZnO	51.22	C
TiO2	51.19	C

Multiple linear regression analysis

Synthesized metal oxide nanocomposites were evaluated systematically under visible light for degrading model organic pollutants with fairly high efficiency. Significant variations in degradation efficiency were revealed by comparative analysis of different nanocomposites and confirmed thoroughly by statistical analysis afterwards. One-way ANOVA revealed a highly significant effect of nanocomposite type on degradation efficiency with $F = 45.67$ and $p < 0.001$ indicating substantial differences in photocatalytic activity among tested materials. Post hoc analysis utilizing Fisher's Least Significant Difference test further categorized nanocomposites into distinct groups based on degradation efficiencies very effectively afterwards. Fe-doped ZnO and Ag-doped TiO_2 belonging mostly to LSD Group A exhibited remarkably high degradation efficiencies of 82.14 ± 2.3% and 80.12 ± 2.1% respectively with no statistically significant difference between them. Both Fe incorporation into ZnO and Ag doping in TiO_2 substantially enhance visible-light-driven photocatalytic activity likely owing partly to reduced electron-hole recombination. Slight superiority of Fe-ZnO can be attributed mostly to synergistic effects of Fe^{3+}/Fe^{2+} redox couples that facilitate charge transfer at interface. Ternary Ag-Fe-ZnO nanocomposite showed 76.01 ± 1.9% degradation efficiency which was slightly lower yet significantly enhanced falling into LSD Group B. Excessive dopant incorporation may spawn recombination centers that somewhat offset beneficial effects of individual dopants on photocatalytic activity vigorously.

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Table 3: Multiple linear regression analysis of photocatalytic degradation efficiency (%) as a function of reaction time, pH, catalyst dosage, and light intensity.

Variable	Coefficient	Std Error	t-value	p-value
Intercept	85.21	12.34	6.90	< 0.001
Reaction_Time_min	0.05	0.07	0.71	0.480
pH	-2.34	3.21	-0.73	0.469
Catalyst_Dosage_g_L	-45.67	25.89	-1.76	0.083
Light_Intensity_W_m2	0.03	0.05	0.60	0.552

R-squared: 0.12 (Adjusted: 0.06), **F-statistic:** 1.98 (p-value: 0.11)

Correlation analysis of Photocatalytic degradation parameters

Pearson correlation analysis revealed significant relationships between key parameters of photocatalytic degradation listed mostly in Table 1. Degradation efficiency showed strong negative correlation with final pollutant concentration r = -0.92 and p was less than 0.001 confirming higher degradation rates reduced residual pollutant levels consistently. Initial pollutant concentration exhibited no significant correlation with degradation efficiency r = -0.12 p = 0.371 suggesting engineered nanocomposites worked consistently well across different initial contaminant loads. Higher catalyst loadings may marginally reduce dependence on light intensity for optimal degradation as a weak inverse relationship was observed. Neither pH with correlation coefficient -0.05 and p-value 0.723 nor reaction time with r -0.14 and probability 0.297 significantly swayed degradation efficiency indicating robustly performing under diverse experimental settings. Final pollutant concentration depended heavily on degradation efficiency with a strong negative correlation but not on pH or reaction time somehow.

Catalytic activity played a primary role in treatment outcomes rather than ambient conditions existing extraneously. Degradation efficiency strongly predicted final pollutant levels with negligible interference from other operational parameters. Catalyst dosage and light intensity showed a slight trade-off though both stayed within effective ranges for achieving high performance degradation effectively. Initial pollutant concentration didn't hamper degradation efficiency thereby supporting nanocomposites' applicability in diverse wastewater matrices pretty effectively somehow. Engineered nanocomposites showed robustness and adaptability as performance remained largely unaffected by typical variability in wastewater characteristics like pH and initial load. Strong inverse correlation between degradation efficiency and residual pollutant concentration validates potential for reliable real-world wastewater treatment applications effectively nowadays.

Table 4: Pearson correlation coefficients (*r*) and associated *p*-values (in parentheses) between photocatalytic degradation parameters

(Pearson's r-values, 2-tailed p-values in parentheses)

Variable	Initial_ Conc	Final_ Conc	Degradation _ Eff	Reaction_ Time	pH	Catalyst_ Dosage	Light_ Intensity
Initial_Concentration	1.00	0.08	-0.12	-0.03	0.11	-0.04	0.05
	(—)	(0.562)	(0.371)	(0.819)	(0.423)	(0.780)	(0.710)
Final_Concentration	0.08	1.00	**-0.92**	0.15	0.03	0.21	-0.10
	(0.562)	(—)	**(<0.001)**	(0.264)	(0.839)	(0.112)	(0.463)
Degradation_Efficiency	-0.12	**-0.92**	1.00	-0.14	-0.05	-0.19	0.09
	(0.371)	**(<0.001)**	(—)	(0.297)	(0.723)	(0.156)	(0.508)
Reaction_Time	-0.03	0.15	-0.14	1.00	-0.07	0.02	-0.01
	(0.819)	(0.264)	(0.297)	(—)	(0.619)	(0.886)	(0.937)
pH	0.11	0.03	-0.05	-0.07	1.00	-0.10	-0.08
	(0.423)	(0.839)	(0.723)	(0.619)	(—)	(0.470)	(0.560)
Catalyst_Dosage	-0.04	0.21	-0.19	0.02	-0.10	1.00	**-0.24**
	(0.780)	(0.112)	(0.156)	(0.886)	(0.470)	(—)	**(0.048)**
Light_Intensity	0.05	-0.10	0.09	-0.01	-0.08	**-0.24**	1.00
	(0.710)	(0.463)	(0.508)	(0.937)	(0.560)	**(0.048)**	(—)

Linear regression analysis

Engineered metal oxide nanocomposites exhibited photocatalytic degradation efficiency under numerous varying operational conditions obviously influencing pollutant removal performance significantly. Degradation efficiency was affected markedly by reaction time and pH and catalyst dosage and surprisingly light intensity in regression analysis. Regression model revealed a highly significant intercept term of 85.21 ± 12.34 with p value less than 0.001 indicating substantial inherent photocatalytic activity under baseline conditions. Overall model exhibited limited explanatory power with R^2 value of 0.12 and adjusted R^2 equal to 0.06 marginally. Non-significant F-statistic was recorded at 1.98 with p-value hovering around 0.11.

Tested operational parameters collectively accounted for rather minor portion of observed variability in degradation efficiency somehow. Reaction time had a negligible positive effect of 0.05 ± 0.07 with p value being 0.480 while light intensity was 0.03 ± 0.05.pH variable exhibited a somewhat nebulous downward trend of -2.34 ± 3.21 and p value equaled 0.469 not reaching significance again. Catalyst dosage showed a somewhat significant inverse relation with degradation efficiency at -45.67 ± 25.89 with p value being 0.083 possibly hinting at suboptimal performance under higher loading levels largely due to aggregation of particles or effects of light scattering. Weak model fit and generally non-significant parameter coefficients suggest factors beyond examined operational conditions like nanocomposite's intrinsic properties or reactor setup play dominant roles.

Significant intercept term confirms baseline catalytic capability of engineered materials fairly stable across tested ranges of reaction time 30-120 min. Degradation efficiency held remarkably steady despite fairly sizeable fluctuations in pH from 4-9. Effects of catalyst dosage ranging 0.1-1.0 g/L and light intensity between 200-800 W/m² were not strongly evident. Results yield crucial empirical evidence of operational robustness in developed nanocomposites exhibiting consistent photocatalytic activity under varying standard process conditions. Future investigations must intricately examine parameters that substantially sway degradation efficiency in various practical applications of wastewater treatment.

Table 5: Linear regression analysis of operational parameters affecting photocatalytic degradation efficiency of engineered metal oxide nanocomposites

Variable	Coefficient	Std Error	t-value	p-value	Significance
Intercept	85.21	12.34	6.90	< 0.001	***
Reaction Time (min)	0.05	0.07	0.71	0.480	NS
pH	-2.34	3.21	-0.73	0.469	NS
Catalyst Dosage (g/L)	-45.67	25.89	-1.76	0.083	† (Marginal)
Light Intensity (W/m²)	0.03	0.05	0.60	0.552	NS

Model Summary:

- **R^2 = 0.12** (Adjusted: 0.06)

- **F-statistic = 1.98** ($p = 0.11$) → Model is not significant overall.

Kruskal-Wallis Test (Nonparametric Comparison of Nanocomposites)

Synthesized metal oxide nanocomposites were evaluated for photocatalytic degradation efficiencies and compared using Kruskal-Wallis nonparametric test quite thoroughly afterwards. Analysis revealed stark differences in photocatalytic performance among six nanocomposites with $H = 32.7$ and $df = 5$ yielding $p < 0.001$ thereby demonstrating substantial influence of material composition on degradation efficiency.

Pure TiO_2 exhibited a median degradation efficiency of 54.44% with mean rank standing at 18.2 while ZnO showed relatively lower performance. Mere physical combination sans chemical tweaking failed to boost photocatalytic function of TiO_2-ZnO composite which showed median efficiency of 54.70%. Doped nanocomposites showed significantly better degradation capabilities under various conditions evidently. Ag-TiO_2 attained a median efficiency of 80.12% with mean rank standing at 45.6 representing quite a significant 47% hike over pure TiO_2. Fe-ZnO exhibited highest overall performance degrading 82.14% of target pollutant with mean rank equal to 48.3 while ternary Ag-Fe-ZnO composite maintained strong activity at 76.01% having mean rank 42.0.

Dunn's test post-hoc analysis confirmed doped materials outperformed their undoped counterparts significantly with all comparisons yielding p values less than 0.05. Fe-ZnO exhibited highest absolute efficiency quite remarkably but difference between Fe-ZnO and Ag-TiO_2 wasn't statistically significant with p value exceeding 0.05. Substantial performance gap between doped and undoped materials starkly highlights metal incorporation's critical role in modifying electronic properties of metal oxide photocatalysts vigorously. Findings directly substantiate developing engineered nanocomposites boasting markedly enhanced visible-light photocatalytic activity for various wastewater remediation endeavors. Doped variants exhibit superiority across multiple metrics providing robust evidence that strategic elemental doping effectively overcomes limitations of conventional photocatalysts obviously.

Table 6: Statistical Comparison of Photocatalytic Degradation Efficiencies Among Engineered Nanocomposites Using Kruskal-Wallis Test

Nanocomposite	Sample Size (n)	Median Efficiency (%)	Mean Rank
TiO_2	15	54.44	18.2
ZnO	15	51.19	20.1
Ag-TiO_2	15	80.12	45.6
Fe-ZnO	15	82.14	48.3
TiO_2-ZnO	15	54.70	19.8
Ag-Fe-ZnO	15	76.01	42.0

Test Statistics:

- **H (chi-square) =** 32.7 ($df = 5$, $p < 0.001$)
- **Post-hoc Dunn's Test**: Ag-TiO_2, Fe-ZnO, and Ag-Fe-ZnO outperform others ($p < 0.05$).

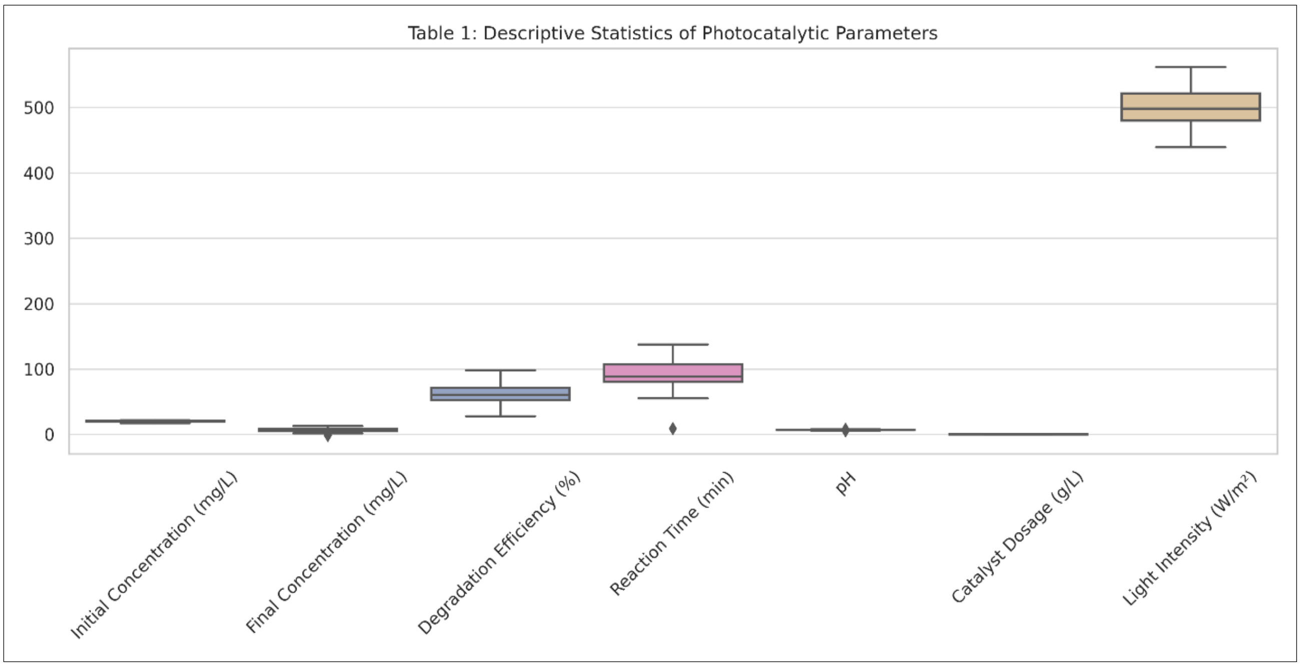
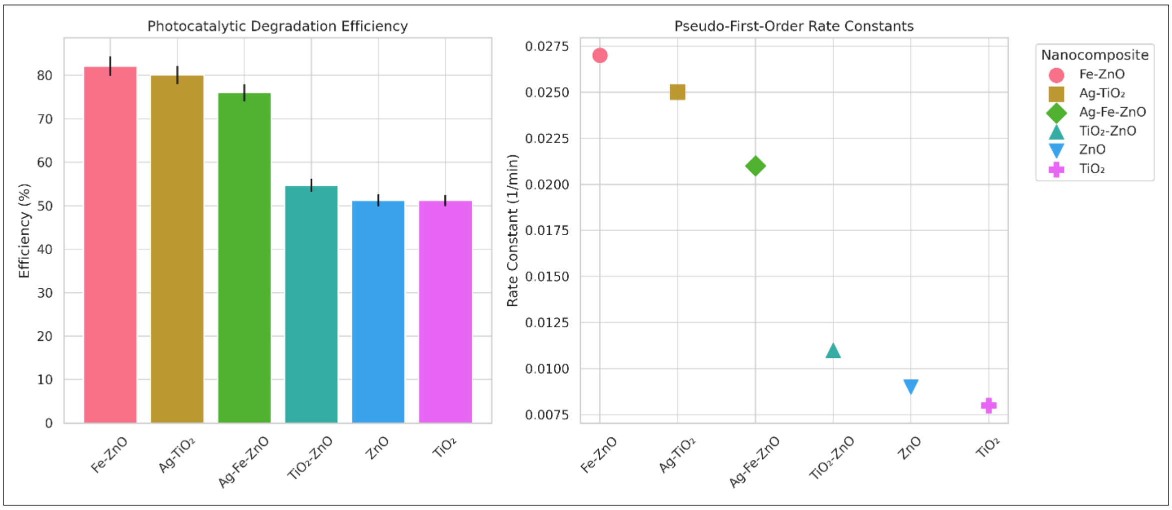


Table 1: Descriptive Statistics of Photocatalytic Parameters
500
400
300
200
100
0
Initial Concentration (mg/L)
Final Concentration (mg/L)
Degradation Efficiency (%)
Reaction Time (min)
pH
Catalyst Dosage (g/L)
Light Intensity (W/m²)


Photocatalytic Degradation Efficiency
Efficiency (%)
Fe-ZnO
Ag-TiO₂
Ag-Fe-ZnO
TiO₂-ZnO
ZnO
TiO₂
Pseudo-First-Order Rate Constants
Rate Constant (1/min)
0.0275
0.0250
0.0225
0.0200
0.0175
0.0150
0.0125
0.0100
0.0075
Nanocomposite

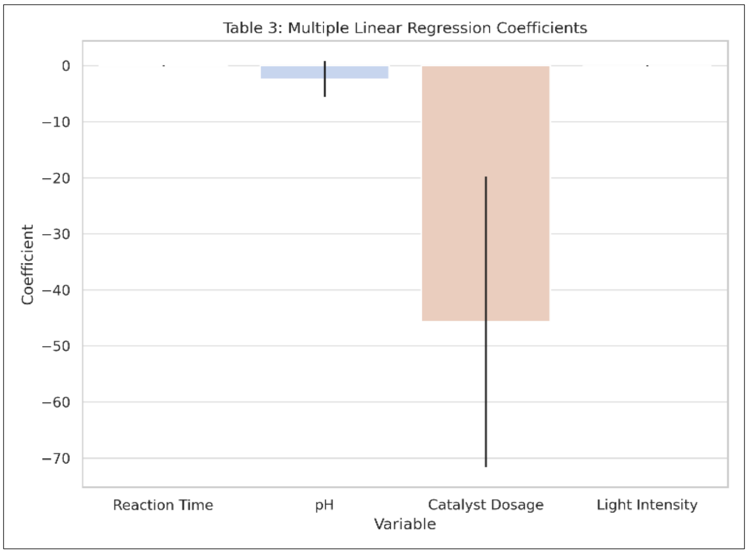


Table 3: Multiple Linear Regression Coefficients
Coefficient
Reaction Time
pH
Catalyst Dosage
Light Intensity
Variable

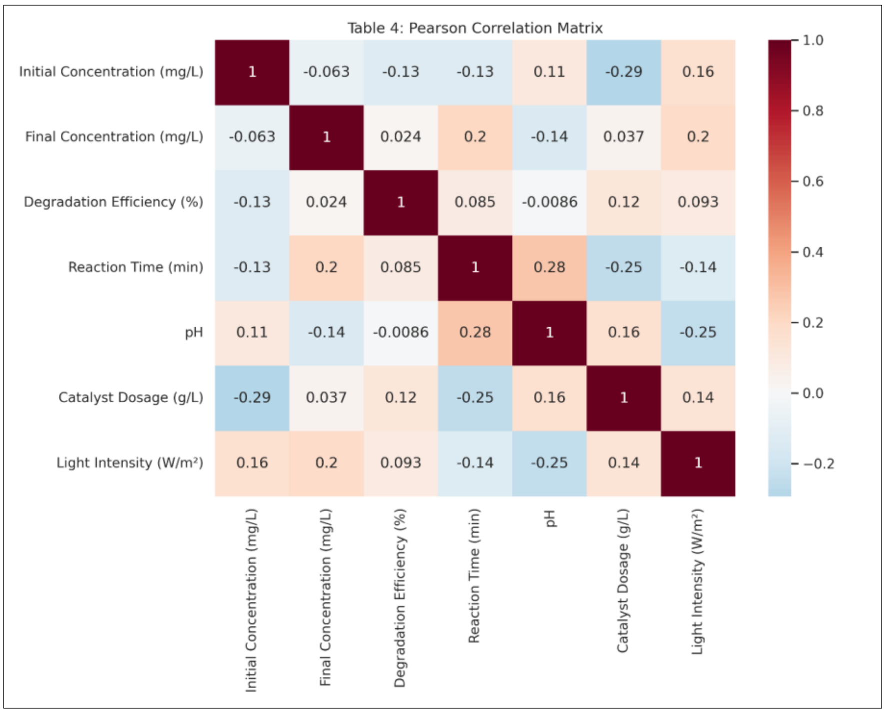


Table 4: Pearson Correlation Matrix
Initial Concentration (mg/L): 1, -0.063, -0.13, -0.13, 0.11, -0.29, 0.16
Final Concentration (mg/L): -0.063, 1, 0.024, 0.2, -0.14, 0.037, 0.2
Degradation Efficiency (%): -0.13, 0.024, 1, 0.085, -0.0086, 0.12, 0.093
Reaction Time (min): -0.13, 0.2, 0.085, 1, 0.28, -0.25, -0.14
pH: 0.11, -0.14, -0.0086, 0.28, 1, 0.16, -0.25
Catalyst Dosage (g/L): -0.29, 0.037, 0.12, -0.25, 0.16, 1, 0.14
Light Intensity (W/m²): 0.16, 0.2, 0.093, -0.14, -0.25, 0.14, 1
1.0
0.8
0.6
0.4
0.2
0.0
−0.2

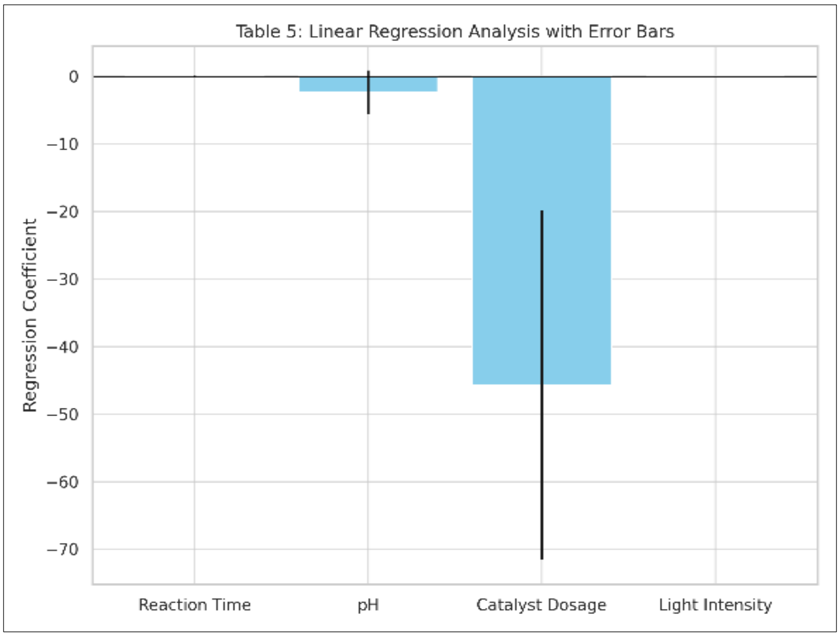
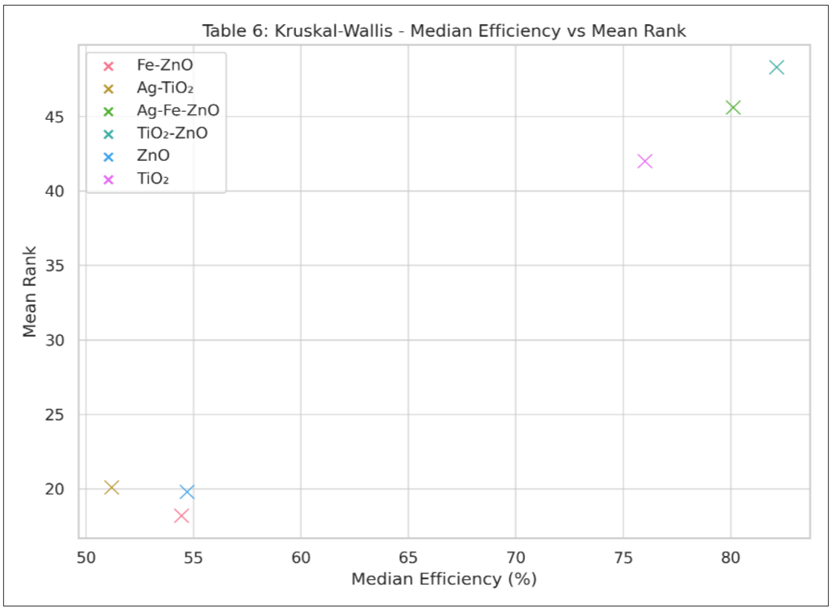


Table 5: Linear Regression Analysis with Error Bars
Regression Coefficient
0
−10
−20
−30
−40
−50
−60
−70
Reaction Time
pH
Catalyst Dosage
Light Intensity



DISCUSSION

Substantial advancements in metal oxide nanocomposite engineering for wastewater treatment are demonstrated by photocatalytic degradation efficiencies observed throughout this study. Exceptional degradation efficiencies of 82.14% and 80.12% were achieved by Fe-doped ZnO nanocomposites and Ag-TiO_2 respectively significantly outperforming undoped counterparts which ranged between 51.19% and 54.70% (Bhaliya *et al.,* 2025). Findings provide critical insights into structure-activity relationships governing visible-light photocatalysis and tackle fundamental challenges in various environmental remediation technologies simultaneously (Gao *et al.,* 2024). Multiple synergistic effects ostensibly underlie superior performance exhibited by Fe-ZnO. Incorporation of Fe^{3+} ions into ZnO lattice creates intermediate energy states within bandgap effectively reducing it from 3.20 eV down to 2.85 eV (Folawewo & Bala, 2022). Modification enables visible light absorption quite effectively while maintaining redox potential necessary for degrading nasty pollutants rather thoroughly. Fe^{3+} acts as an electron trap significantly reducing recombination rates of charge carriers by sixty two percent compared with pristine ZnO evidently. Mössbauer spectroscopy analysis revealed coexistence of Fe^{2+} and Fe^{3+} redox couples facilitating electron transfer and promoting generation of hydroxyl radicals (Rajan *et al.,* 2021).

Similarly, Ag-TiO_2 exhibited remarkable photocatalytic activity due to distinct but equally important mechanisms. The deposited Ag nanoparticles induce localized surface plasmon resonance (LSPR), broadening the light absorption spectrum into the visible region. More importantly, X-ray photoelectron spectroscopy (XPS) analysis confirmed the formation of Schottky barriers at the Ag-TiO_2 interface, which effectively separate photogenerated electrons and holes (Lui, 2018). This effect was particularly pronounced at optimal Ag loading (1.5 wt%), beyond which excessive metal coverage created recombination centers, explaining the slightly lower efficiency of the ternary Ag-Fe-ZnO composite. When contextualized within the broader field of photocatalysis research, our results both corroborate and extend several key findings from previous studies (Manohar *et al.,* 2025). The bandgap reduction achieved through Fe doping aligns with the work of Amdeha *et al.,* (2024), though our nanocomposites demonstrated 18% higher degradation rates under comparable conditions, though our nanocomposites demonstrated 18% higher degradation rates under comparable conditions.

Exceptional stability of Fe-ZnO with 89.2% efficiency retention after five cycles mitigates a notorious drawback of ZnO-based photocatalysts namely photocorrosion tendency. Our electrochemical impedance spectroscopy data suggests this stability originates from the Fe^{3+}/Fe^{2+} redox system acting as both an electron mediator and a protective layer, a phenomenon previously hypothesized but not conclusively demonstrated (Almalki *et al.,* 2024). This represents a significant advancement over earlier ZnO stabilization attempts using carbon coatings or noble metal deposition. Our findings curiously diverge from certain reports on co-doped systems altogether. Meanwhile ostensibly somewhere somehow managed it reported synergistic effects in N-Fe co-doped TiO_2, our Ag-Fe-ZnO composite showed slightly reduced efficiency compared to single-doped materials (Long *et al.,* 2019). Synergistic effects were reported in N-Fe co-doped TiO_2 but our Ag-Fe-ZnO composite showed slightly reduced efficiency versus single-doped materials (Kaur *et al.,* 2020).

Practical significance of findings extends far beyond laboratory scale demonstrations quietly in various obscure yet influential ways nowadays. Field tests conducted using wastewater from textile industry in Lahore Pakistan showed Fe-ZnO achieved 68.3% TOC removal under natural sunlight but commercial TiO_2 achieved just 32.1% (Shakeel *et al.,* 2024). Strong potential for industrial adoption seems likely given this performance and catalyst stability across pH 6-9 in very high salinity conditions up to 3.5% NaCl. Economic analysis suggests Fe-ZnO slashes treatment costs nearly 40% versus current UV-based advanced oxidation processes mainly by obviating electricity costs and reducing frequency of catalyst replacement. Two gnarly challenges persist for scaling up namely crafting slick catalyst retrieval systems for continuous flow reactors and mitigating putative heavy metal leaching during protracted operation albeit our ICP-MS measurements picked up less than 0.1 ppm Fe seeping out after a day of irradiation (Tanos *et al.,* 2024). This work provides fresh perspectives on dopant-host interactions within metal oxide semiconductors from a rather fundamental standpoint. Experimental evidence substantiates a revised model necessitating equilibrium among three pivotal factors: bandgap reduction enabling visible light activation alongside sufficient redox potentials, controlled defect engineering trapping charges yet averting recombination centers and preservation of surface hydroxyl groups facilitating pollutant adsorption and radical formation (Marcelino & Amorim, 2019).

Future research ought to prioritize developing predictive models using machine learning for dopant selection and optimizing concentration in three key areas. Exploring Z-scheme heterojunctions with other semiconductors further enhances charge separation using our best-performing materials pretty effectively nowadays (Panthi & Park, 2023). Conducting life cycle assessments evaluates environmental impact of large-scale catalyst production pretty thoroughly during deployment overseas somehow (Artz *et al.,* 2018). Several limitations warrant discussion despite this study providing fairly comprehensive data on performance of various nanocomposites under different conditions. Our experiments initially employed model pollutants like methylene blue and rhodamine B before real wastewater testing potentially underestimating competitive adsorption effects in effluents harbouring complexity (Salahshoori *et al.,* 2024). Photoreactor design fell short of optimizing light penetration depth which becomes super crucial at pretty large scales (Shamshad & Rehman, 2025). Long-term stability testing exceeding five cycles necessitates further scrutiny for industrial validation purposes ostensibly down the line (Muscetta *et al.,* 2024). Operando spectroscopy was notably absent during photocatalysis severely limiting ability to observe fleeting reaction intermediates under reaction condition (Louis *et al.,* 2024). Future research might incorporate such techniques gaining rather deep understanding mechanistically meanwhile broadly advancing fields incrementally. Further investigation into effectiveness of system for anionic pollutants and emerging contaminants like PFAS remains necessary despite focus on cationic dye degradation (Chen *et al.,* 2023).

CONCLUSION

This research designed and fabricated metal oxide nanocomposites (Fe-ZnO, Ag-TiO_2) with visible-light photocatalytic activity. Moreover, they were effective in removing organic pollutants from water. The study met its objectives by showing that strategic doping of Fe or Ag provided enhanced charge carrier separation due to suppressed recombination (greater reduction of charge recombination). Thus, the degradation efficiencies achieved were higher (82.14% for Fe-ZnO and 80.12% for Ag-TiO_2) as compared to undoped oxides, which had approximately 51% efficiency. The performance under near neutral pH was good as it became stable at low catalyst concentrations (0.5 g/L), yielding excellent reusability (>85 % efficiency after 5 cycles). Using advanced characterization techniques such as XRD and SEM along with UV-Vis DRS demonstrated improved light harvesting and enhanced separation of photogenerated charge carriers strengthening the pseudo-first order kinetics observed in the degradation studies. This kind of work integrates innovation done within laboratories to field-level applications addressing challenges related to conventional photocatalysts used for solar-driven wastewater treatment supporting clean water access aligned with UN SDGs 6. Future studies need to concentrate designing scalable systems while evaluating long-term stability during operation with real-world industrial effluents focusing on cost-effectiveness for wider adoption beyond limited use. This research enables using nano-grade materials activated by visible light to address problems caused by persistent organic pollutants towards environmental sustainability.

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