

Research Article

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Fe-doped ZIF-8 for sunlight-driven photocatalytic degradation of methylene blue: synthesis, characterization, and AI-based process modeling

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Abstract: The growing environmental cost of wastewater dye effluents is reason enough to adopt effective, sustainable remediation measures. This paper reports a new, environmentally friendly synthesis of bimetallic $\text{Fe}_x\text{Zn}_{1-x}\text{ZIF-8}$ structures through a fast, solvent-free, room-temperature mechanochemical synthesis without using toxic solvents or high energy expenditures. The $\text{Fe}_{0.075}\text{Zn}_{0.925}\text{-ZIF-8}$ has much higher visible-light photocatalytic activity than pristine ZIF-8, with over 95 % degradation of methylene blue (MB) under solar simulation in 40 min. Successful incorporation of Fe, prolonged absorption in visible light, decreased bandgap (2.8 eV at $x = 0.075$), and inhibited recombination of charges were confirmed by comprehensive characterization (XRD, SEM-EDS, XPS, UV-Vis), which explains the mechanism contributing to improved performance. More importantly, this material serves two purposes (1): as a high-efficiency, reusable photocatalyst that takes advantage of synergistic radical-mediated dye degradation using Fe/Zn sites and (2) as a model of the framework of an artificial intelligence (AI) model. The experimental degradation data were trained on machine learning algorithms (Artificial neural networks) using the radiation time and the catalyst dosage. The AI models demonstrated high predictive accuracy ($R^2 = 0.995$, RMSE = 0.005) that allowed the optimization of the degradation conditions with high precision and the

demonstration of the non-linearity among the parameters. This study is the first to bridge sustainable materials design and AI-based process engineering to provide a scalable, environmentally friendly solution to the dye wastewater treatment problem and an effective predictive toolkit to drive the development of a photocatalytic system.

Keywords: metalorganic framework; photocatalysis; MB; artificial intelligence; ANN

1 Introduction

The worsening scenario of water pollution caused by the existence of industrial effluents that contain non-biodegradable organic dyes is a serious menace to the ecosystems and the health of humans [1, 2]. One of such contaminants is the widely used and nonbiodegradable methylene blue (MB), which requires the creation of innovative and efficient treatment methods [3, 4]. One of the solutions has become semiconductor-based photocatalysis, which utilizes light energy to produce reactive oxygen species, which can further mineralize complex organic molecules to non-toxic end products [5, 6]. The search for a perfect photocatalyst, efficient, stable, cost-efficient, and working under intense sunlight, is still an important challenge, and the research of new material architecture is encouraged.

Metal-organic frameworks (MOFs) represent a novel class of porous materials that have acquired significant consideration in recent years owing to their prospective applications in gas storage and separation, catalysis, and templating [7, 8]. MOFs belong to a larger family of compounds called metalorganic frameworks: zeolite imidazolate frameworks (ZIFs) consist of transition metal ions, [Zn(II), Co(II)], and imidazolate linker, which form the three-dimensional tetrahedral arrangements [9, 10]. In the recent past, ZIFs have garnered a lot of attention due to their unparalleled and eminently suitable characteristics, like large external surface area, crystallinity, and thermal/chemical sustainability [11, 12]. Of these, ZIF-8 [Zn(2-

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methylimidazole)₂·2H₂O], also known as Zn(II) imidazolate, built from 2-methylimidazole ligands and Zn(II) ions, shows higher thermal and chemical stability than other MOFs. That is why it includes a sodalite substructure, with the internal porosity irradiated by openings of 0.34 nm.

Nonetheless, the ultraviolet range, the only portion of the solar spectrum that pristine ZIF-8 can absorb, is presented in a wide bandgap and thus represents a very low percentage of the solar spectrum, thereby limiting its practical use in natural sunlight [13, 14]. To address this drawback, transition metal strategic doping of ZIF-8, e.g., with iron (Fe), has been suggested as an efficient approach to visible-light sensitization of ZIF-8. Although a couple of studies have been conducted on Fe-doped ZIF-8 (Fe-ZIF-8) to apply in varied applications, a direct and systematic comparative study indicates clear benefits of a material synthesized by us. As an example, Fe-ZIF-8 can provide a superior compromise between high visible light absorption and aqueous degradation relative to other MOFs such as MIL-100(Fe) (which can be hydrolytically unstable) and benchmark photocatalysts such as TiO₂ (which is not only mainly UV-active, but can also be hydrolytically unstable) [15]. Moreover, the Fe-doping is especially beneficial in comparison with other doped ZIF-8 systems (e.g., Co or Cu) because of its low cost, non-toxicity, and various charge-transfer pathways resulting in the use of the best photocatalytic performance [16, 17]. Although these are promising characteristics, most studies that have been reported on using Fe-ZIF-8 as a photocatalyst use high-power artificial sources of UV-visible light. There is a straw-man gap in the overall assessment of its performance with realistic irradiation of sunlight, which is needed to gauge its potential real capacity in solar energy's scalable water decontamination. Furthermore, optimization of the photocatalytic process is regularly done using standard one-variable-at-a-time techniques, which are inefficient and do not reproduce the interactions between parameters.

The proposed study aims to fill these gaps by synthesizing and characterizing the Fe-ZIF-8 systematically, with the aim of using sunlight-driven photocatalytic degradation of MB. The main hypothesis of the study is twofold: to prove quantitatively the superiority of Fe-ZIF-8 in comparison with other widespread photocatalysts under natural sunlight, and to go beyond the process of empirical optimization by creating a powerful, artificial intelligence-based model to predict and optimize the degradation process. The first issue will be addressed in this work with the help of the comparison of the photocatalytic activity of pristine ZIF-8 with pristine ZIF-8 and other reference materials. To deal with the second, we shall use machine learning models, such as artificial neural networks (ANN) and optimization strategies

to model the complicated nonlinear relationships among key process variables (e.g., catalyst dosage and irradiation time) and degradation efficiency. Although the present work represents a major contribution to the field of solar photocatalysis and process simulation, it also has its flaws; the authors concentrate on one model pollutant (MB) and do not elaborate on the impact of natural water matrices and the recyclability of catalysts on a long-term basis. These areas, combined with the extension of the AI model to a larger portion of variables and other types of pollutants, are found to be the main trends of further research, as they will allow turning this technology out of the laboratory setting and into practice.

2 Materials and methods

2.1 Materials

Zn(NO₃)₂·6H₂O (Loba Chemie Pvt. Ltd., India), 2-methylimidazole dispersed (Loba Chemie Pvt. Ltd., India), TEA (Sigma Aldrich, USA), Methlen Blue (Loba Chemie Pvt. Ltd., India).

2.2 Synthesis of Fe-doped ZIF-8

A modified synthesis approach was utilized for producing ZIF-8 crystals. This standard synthesis included a solution of 0.00675 mol Zn(NO₃)₂·6H₂O (Loba Chemie Pvt. Ltd., India) entirely dissolved in 0.015 L demineralized water (DW), together with a solution containing 0.054 mol 2-methylimidazole dispersed (Loba Chemie Pvt. Ltd., India) in 15 mL DW and 0.0078 L TEA (Sigma Aldrich, USA) [18]. The solutions mixed together underwent continuous stirring for 1 h before the mixture was aged for 24 h at room temperature. The white product required three sequential washes with 0.03 L solutions of DW and two identical washes with 0.03 L methanol solutions. Researchers dried and labelled their ZIF-8 product at 60 °C for 6 h [19].

2.3 Materials characterization

The structural, morphological, and elemental composition of the prepared powder was analyzed using X-ray diffraction (LANScientific) and transmission electron microscopy [JSM-IT200 (JEOL)] equipped with energy-dispersive X-ray spectroscopy (EDX). The optical properties and photocatalytic performance were evaluated using a UV-visible spectrophotometer [4, 20].

2.3.1 ANN method

Here, artificial neural networks (ANNs) were selected for this modeling task due to their proven capability to learn and accurately represent complex, non-linear relationships between input variables and output responses, which are often encountered in photocatalytic processes. Unlike traditional linear regression or other simpler models, ANNs can capture the intricate interactions between parameters like catalyst dosage and irradiation time without requiring a pre-defined mathematical form, making them particularly suitable for optimizing such multi-factor systems [21].

ANN was developed to predict the photocatalytic removal efficiency based on two main variables: doping percentage and irradiation time. The model architecture included several hidden layers using ReLU activation, and training was performed using the Adam optimizer with MSE as the loss function, as shown in Figure 1.

The 87 experimental data points were divided into training, validation, and test subsets, comprising 62, 7, and 18 samples, respectively. After training, the model showed strong predictive performance. On the test set, the R^2 value reached **0.995**, which indicates an excellent fit. The calculated metrics were as follows:

Metric	Value
Mean squared error (MSE)	4.427
Root mean squared error (RMSE)	2.104
Mean absolute error (MAE)	1.648
Coefficient of determination (R^2)	0.995

where:

$$MSE = \frac{1}{n} \sum_i^n (y_i - \hat{y}_i)^2 \quad (1)$$

$$RMSE = \sqrt{\frac{1}{n} \sum_i^n (y_i - \hat{y}_i)^2} \quad (2)$$

$$MAE = \frac{1}{n} \sum_i^n |y_i - \hat{y}_i| \quad (3)$$

These results suggest that the model was able to learn the relationship between the input parameters and the experimental efficiency values quite well.

3 Result and discussion

3.1 Structural properties

Figure 2a displays the XRD patterns of undoped ZIF-8 and Fe-doped ZIF-8. The XRD patterns of ZIF-8 align well with those reported in references [22]. A distinct and sharp diffraction peak corresponding to the (111) plane was observed at $2\theta = 7.4^\circ$ in the XRD pattern of ZIF-8, representing high crystallinity. The XRD patterns of Fe-doped ZIF-8 showed typical peaks of ZIF-8, with no detectable peaks corresponding to iron oxide. Nonetheless, the intensity of these diffraction peaks reduced as the amount of incorporated iron increased. However, the increase in the Fe concentrations led to a shift in the patterns further two theta higher, indicating a decrease in the interplanar spacing (d-spacing),

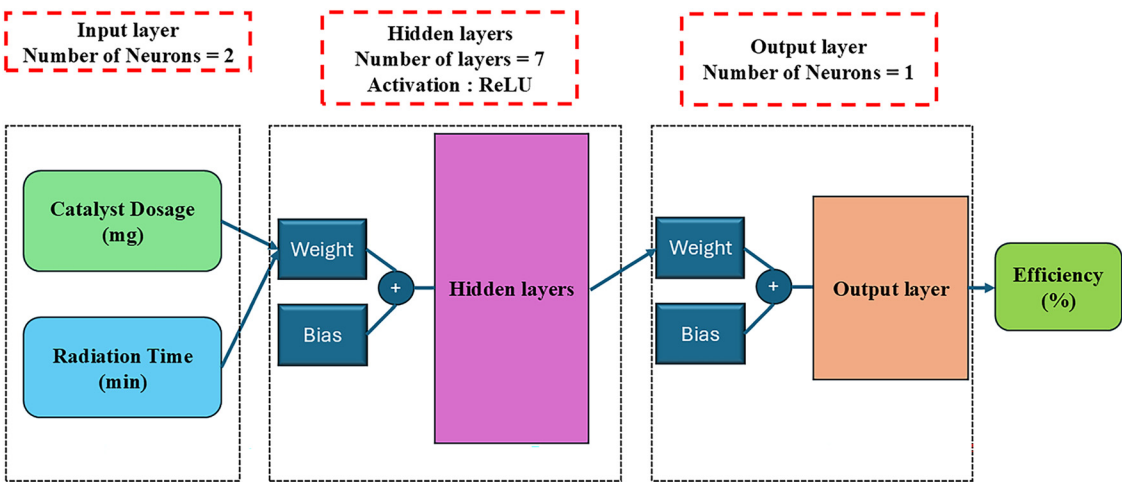


Figure 1: The structure of the used ANN in the present study.

often due to the incorporation of smaller atoms or compressive strain, as shown in Figure 2b. The detection of shifts in XRD peaks would suggest Fe ions entered the ZIF-8 framework, which transformed its lattice parameters.

3.2 Morphological properties

An examination of Fe-ZIF-8 and its derivative with Fe-ZIF-8 alongside standard materials ZIF-8 and ZIF-8 through TEM revealed their morphological aspects and microstructural characteristics, as shown in Figure 3. The Fe-ZIF-8 crystals maintained their rhombohedral dodecahedron shape with standardized dimensions the same as in previously documented studies [23]. The Fe-doped ZIF-8 crystals demonstrated average particle dimensions that were smaller than those of pure ZIF-8 crystals. Execution of Fe ions throughout synthesis resulted in quick nucleation and crystal formation events while directly contributing to ZIF-8 material size diminishment [24].

The surface composition of pure ZIF-8 and Fe-doped ZIF-8 was analyzed with XPS. Figure 4a demonstrates survey spectra that prove the existence of elements Zn, Fe, N, and C in these MOFs. The XPS spectrum of Zn 2p prompts two main peaks found at 1,021.8 and 1,044.8 eV that match Zn 2p_{3/2} and Zn 2p_{1/2} findings documented in Zhou et al. [25]. Data in Figure 4c reveals the N 1s XPS spectrum has three distinct peaks distributed at 398, 399, and 401 eV relating to pyridinic N and Fe–N and pyrrolic N configurations [26].

3.3 Electronic properties

Figure 3d shows the C 1s spectrum of Fe-ZIF-8, with deconvolution analysis identifying three components within the

overlapping peak. The high-resolution C 1s spectrum in Figure 4d can be fitted to three peaks at 285, 286.2, and 288.3 eV, related to C=C/C–C, C=N, and C–N bonds, correspondingly. The existence of the Fe–N peak confirms the successful production of Fe-ZIF-8 [27, 28]. While the carbon surface and the species of nitrogen in ZIF-8 are similar to those in Fe-ZIF-8, the Fe–N peak, absent in ZIF-8, is detected in Fe-ZIF-8, demonstrating the successful inclusion of Fe into the ZIF-8 framework through doping.

The presence of Fe in Fe-ZIF-8 is further supported by the identification of various iron peaks in Figure 4e, including Fe(II) 2p_{3/2} at 710.2 eV, Fe(III) 2p_{3/2} at 713.8 eV, Fe(II) 2p_{1/2} at 723.4 eV, and Fe(III) 2p_{1/2} at 726.7 eV [29]. Additionally, the C and N spectral peaks shift to lower binding energies, which is attributed to changes in the chemical interactions among C, N, Fe, and Zn atoms resulting from Fe incorporation and activation [30].

3.4 Optical properties

Researchers measured synthesized samples' optical absorbance properties using double-beam UV–Vis spectrophotometry to observe photocatalytic performance and optical characteristics as shown in Figure 5a. The absorbance peak at 285 nm originates from scattering behavior, which results from randomly placed grain boundaries and nanocrystallite distribution. The research demonstrated that Fe concentrations led to larger crystallite sizes while producing reductions in the optical bandgap values from 3.6 eV to 2.8 eV, as demonstrated in Figure 5b. The absorbance edge experienced a slight moving toward red wavelengths as the iron levels increased, leading to bandgap narrowing. Fe-doped ZIF-8 shows a reduced bandgap because 3d electrons from Fe(III) or Fe(II) move toward ZIF-8's conduction band

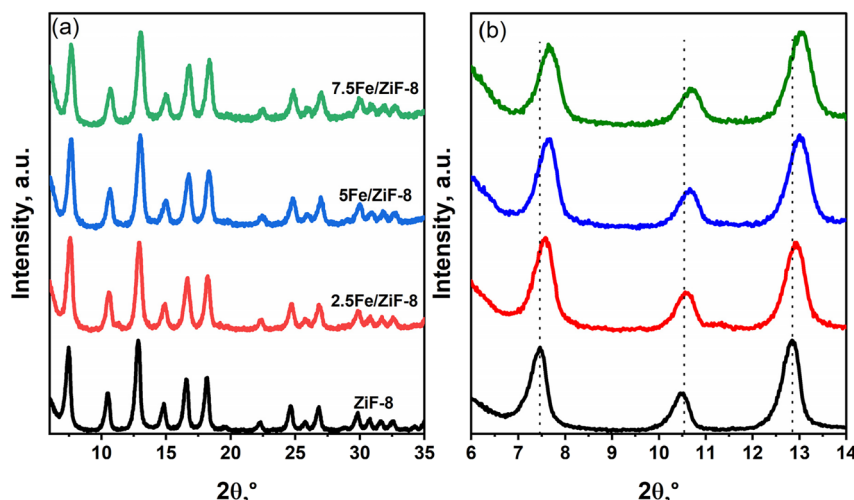


Figure 2: X-ray diffraction analysis of ZIF-8 (a) XRD patterns of several concentrations of iron-doped ZIF-8, (b) selected 2 theta of different concentrations of iron-doped ZIF-8.

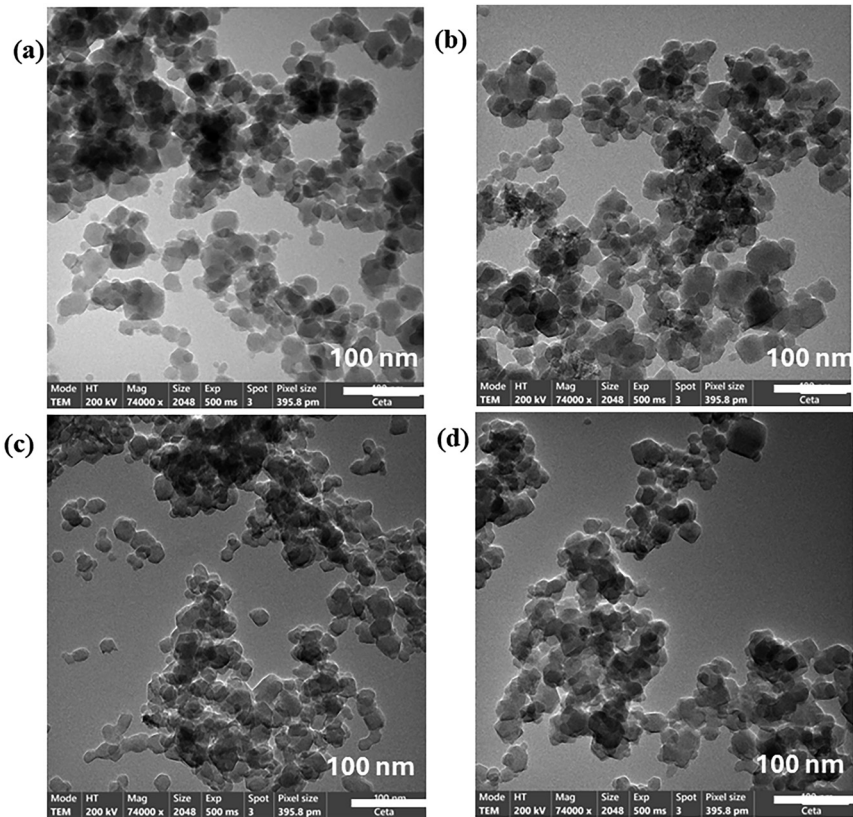


Figure 3: TEM photos of different concentrations of Fe-doped ZIF-8.

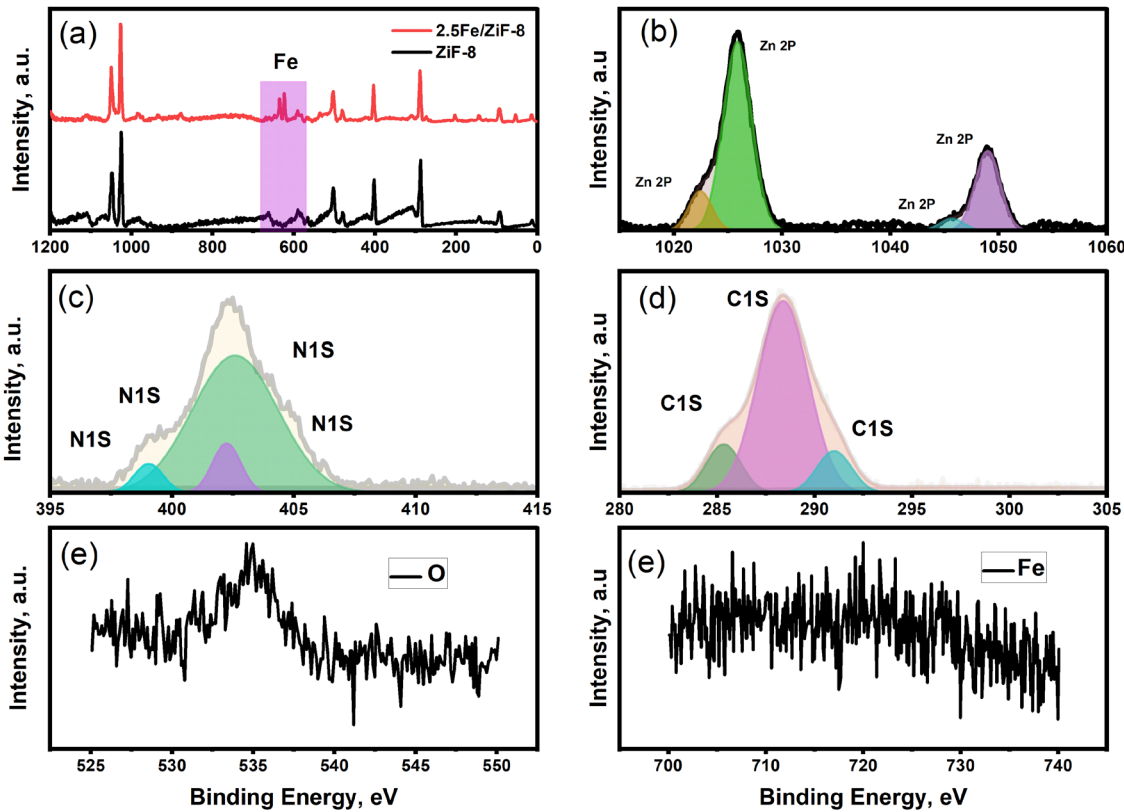


Figure 4: XPS of Fe doped ZIF-8.

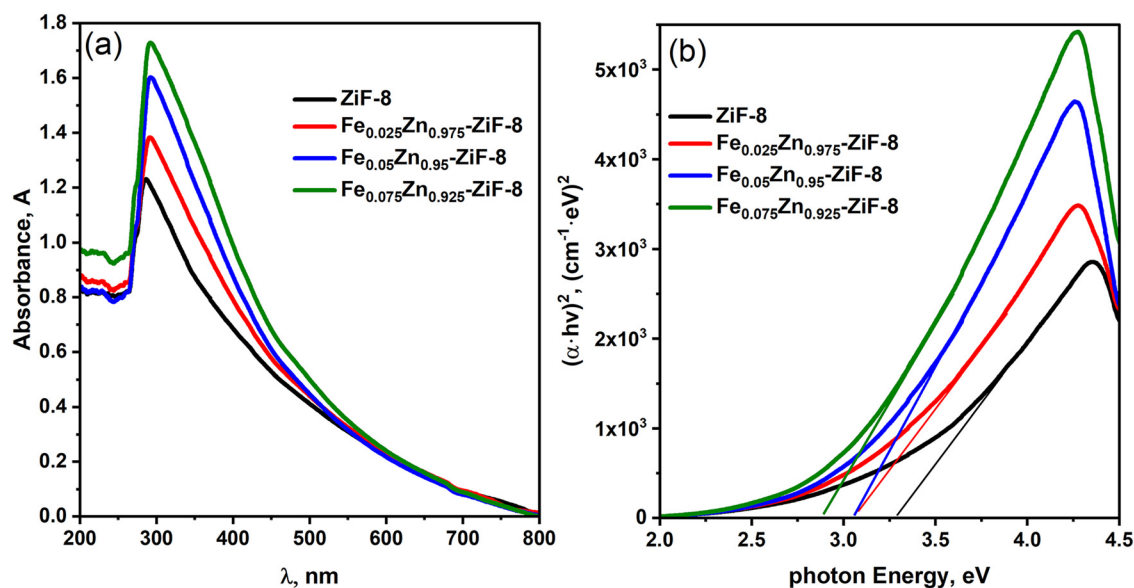


Figure 5: Optical analysis (a) The UV–visible absorbance spectra provide analysis for all produced materials. (b) Tauc plots show energy band gap measurements for all samples.

through a charge transfer transition. Fe-doped ZIF-8 displays visible light photocatalytic behavior according to the research data.

3.5 Photocatalytic activity

Figure 6a–d illustrate the photocatalytic activity of ZIF-8, 0.025, 0.05, and 0.075 Fe-doped ZIF-8 photocatalysts in the MB

dye degradation under the irradiation of visible light. The value of the highest absorbance peak was observed at 662 nm, corresponding to the characteristic peak of MB dye. Notably, the absorbance value of MB dye remained unchanged in the absence of photocatalysts over the investigated time interval, even under visible light illumination. Additionally, undoped ZIF-8 exhibited limited photocatalytic activity under visible light (Figure 6a). The degradation efficiency improved slightly when Fe-doped ZIF-8 was

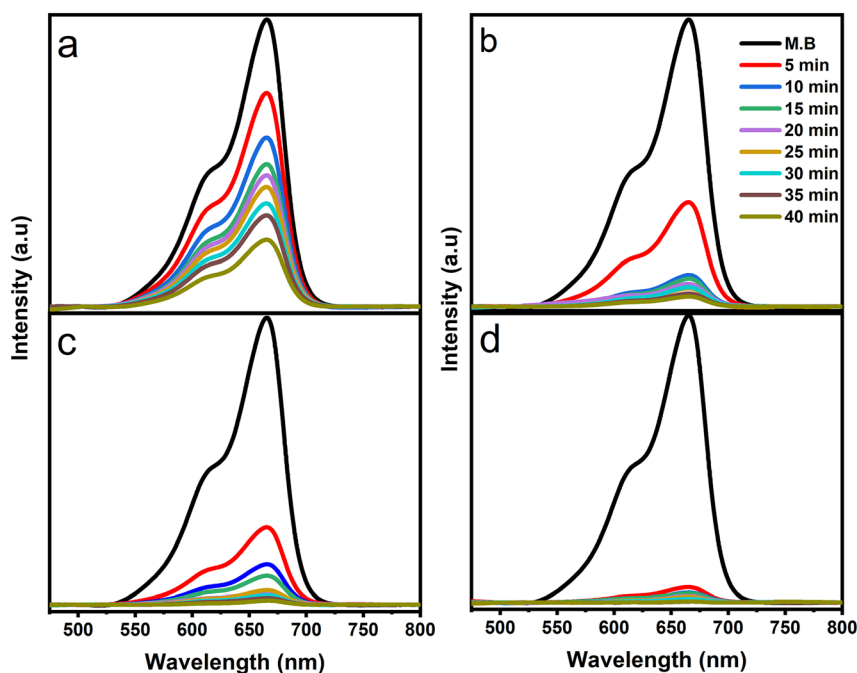


Figure 6: Absorbance spectra of MB dye following wastewater treatment under sunlight irradiation in the presence of photocatalysts: (a) pure ZIF-8, (b) 0.025, (c) 0.05, and (d) 0.075 ($\text{Fe}_x\text{Zn}_{1-x}$ -ZIF-8).

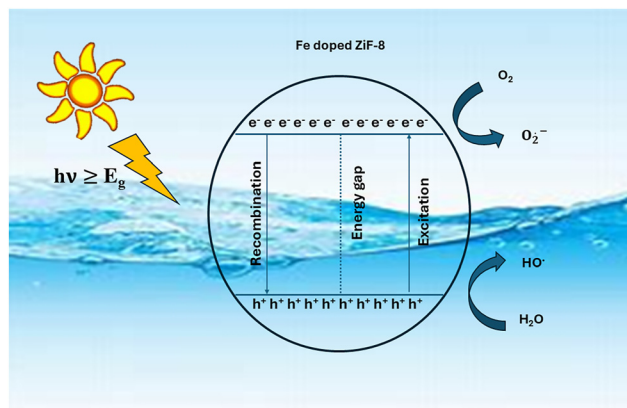
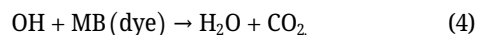


Figure 7: Mechanism of photocatalytic MB dye degradation using the synthesized materials.

operated as the photocatalyst (Figure 5b). This degradation efficiency further increased with higher Fe doping, requiring approximately 40 min to achieve ~96 % degradation (Figure 6c and d).

When sunlight with energy equal to or exceeding the bandgap energy interacts with the photocatalyst surface, pairs of electrons and holes are created, triggering the photocatalytic activity. The valence band holes, together with excited electrons in the conduction band, participate in reactions with O_2 molecules nearby, creating superoxide radical anions while surface-adsorbed water molecules produce hydroxyl radicals ($OH^\cdot$). These free radicals, known as reactive oxygen species (ROS), rapidly react with organic

pollutants, breaking them down. Figure 7 depicts the apparatus for this photocatalytic performance.



After the MB dye completely degrades, the photocatalyst can be recovered by centrifugation from the solution. Figure 8a represents the values of degradation efficiency (C/C_0) that describe MB dye degradation through time. The enhanced photocatalytic performance with growing iron concentration can be attributed to the following factors: The material develops two important effects as a result of the added Fe content: it enhances photon absorption through localized states, and its superior oxygen vacancy concentration minimizes electron-hole recombination. Additionally, the obtained data were used to analyze the degradation kinetics of MB dye using the following pseudo-first-order kinetic equation [31, 32]:

$$-\ln\left(\frac{C_t}{C_0}\right) = kt \quad (5)$$

where C_0 represents the dye preliminary concentration and C_t represents the dye concentration after time irradiation t , and k represents the constant of rate. The analyzed results are displayed in Figure 8b. The data indicate that the degradation process adheres to pseudo-first-order kinetics. The constant of the rate (k) for pure ZIF-8, 0.025, 0.05, and 0.075 Fe-doped ZIF-8 photocatalysts are 0.031, 0.071, 0.073, 0.094, and 0.11 min^{-1} , respectively. These findings demonstrate that the degradation rate is 3.5 times faster for the

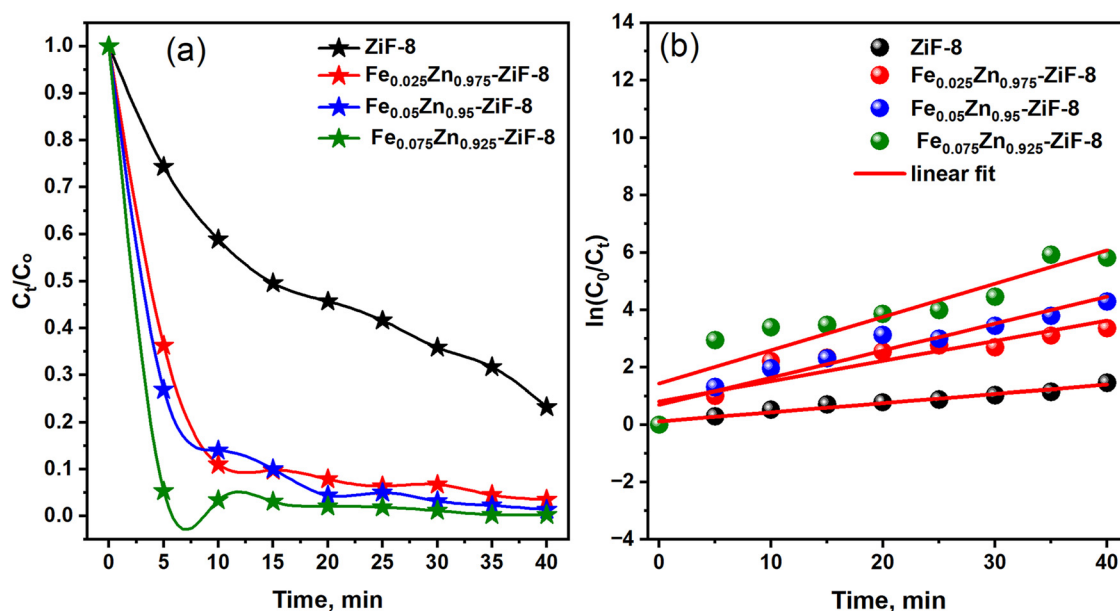


Figure 8: Catalytic investigation of ZIF-8 (a) C/C_0 vs time spectra for ZIF-8 doped with numerous Fe concentrations, (b) Reaction rate constant for the photocatalysts ZIF-8 doped with numerous Fe concentrations.

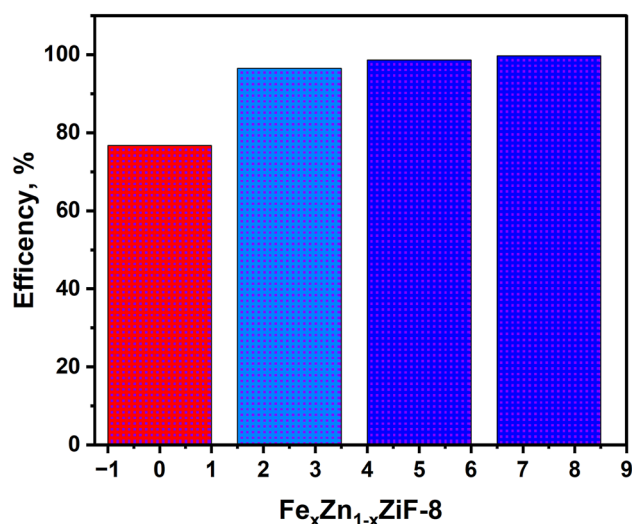


Figure 9: Bar graph illustrating all synthesized materials' degradation percentage versus Fe concentration.

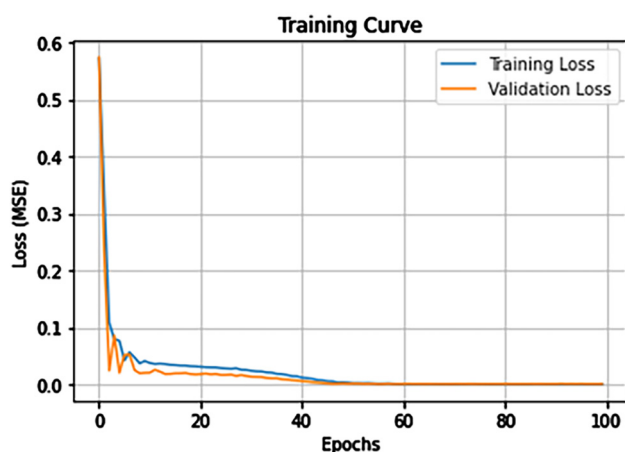


Figure 10: Mean squared loss values during training and validation across 100 epochs.

0.1 Fe-doped ZIF-8 photocatalyst than for the pure ZIF-8 photocatalyst. The notable enhancement in the kinetic constant k is due to the modifications of ZIF-8 achieved through the incorporation of varying concentrations of Fe dopants.

The degradation efficiency is calculated using the following equation [19, 33]:

$$\% \text{ degradation} = \frac{C_0 - C_t}{C_0} \times 100 \% \quad (6)$$

where C_0 and C_t represent the value of the solution absorbance at time 0 and t , individually and is shown in Figure 9.

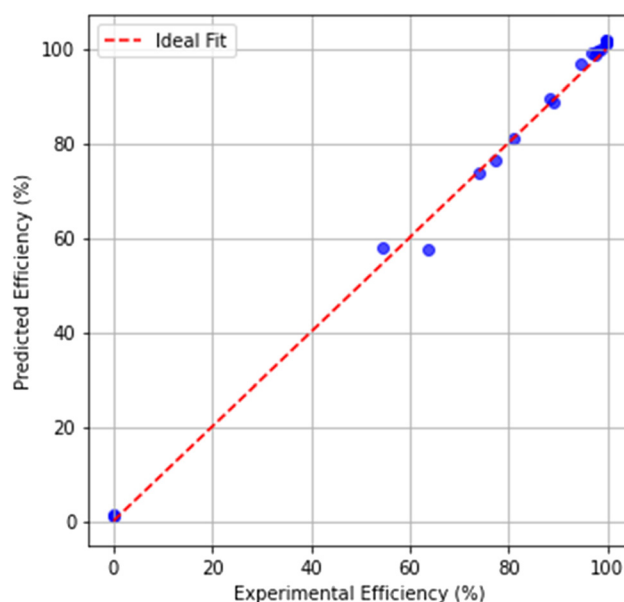


Figure 11: Comparison between predicted and measured efficiency values from the test dataset.

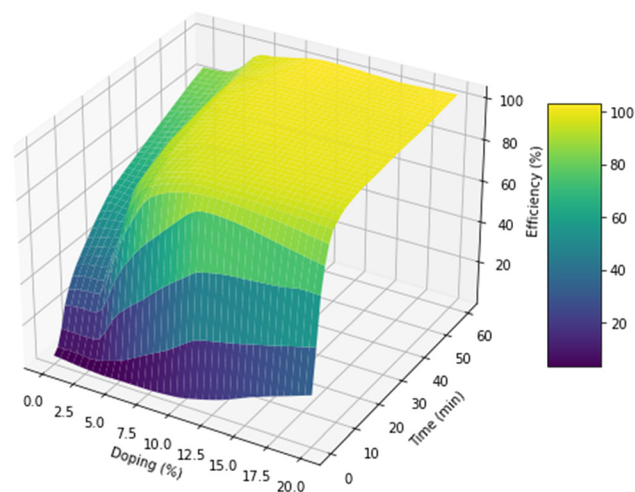


Figure 12: Efficiency as a function of doping level and irradiation time.

3.6 ANN architecture and training methodology

3.6.1 Training behavior

The training progress was tracked over 100 epochs. As shown in Figure 10, both training and validation loss decreased smoothly over time. There was no sign of over-fitting, as the validation curve closely followed the training curve. This suggests the model was able to generalize well to unseen data.

3.6.2 Prediction accuracy

To evaluate how well the model predicts actual experimental outcomes, a scatter plot of predicted versus experimental efficiency values was drawn (Figure 11). The points were tightly clustered around the diagonal ($y = x$), showing strong agreement between the predicted and measured results. This further confirms the reliability of the model.

3.6.3 Surface visualization of model predictions

To better understand how the model captures the interaction between doping percentage and irradiation time, a 3D surface plot was generated (Figure 12). This plot presents the predicted photocatalytic efficiency as a function of the two input variables. As seen, the model predicts a rapid increase in efficiency with both higher doping levels and longer irradiation times, particularly at lower doping values, where efficiency rises steeply. Beyond a certain doping threshold, however, the efficiency reaches an endpoint, indicating diminishing returns with further increases. This behavior is consistent with experimental expectations, where excessive doping can lead to recombination effects or other limiting phenomena.

4 Conclusions

In summary, pure ZIF-8 and different Fe concentrations (0, 0.025, 0.05, and 0.075) based ZIF-8 were successfully synthesized. The XRD confirmed that all samples have the same cubic phase without contamination by another phase. The Fe-doping ZIF-8 brought a red shift, driving the band gap in visible light. The average crystallite size grew with the incorporation of Fe, accompanied by a reduction in the optical bandgap from 3.6 to 2.8 eV. The X-ray photoelectron spectroscopy (XPS) showed Zn 2p peaks at ~1,021 and 1,044 eV for the ZIF-8 and Fe-doped ZIF-8. The Fe-ZIF-8 can act as a sunlight-driven photocatalyst for highly efficient degradation of Mb dye. The photocatalytic performance of $\text{Fe}_{0.075}\text{Zn}_{0.925}$ -ZIF-8 showed optimal results with an observed rate constant of 0.11 min^{-1} , which enabled 96 % MB dye decomposition during a 40-min reaction period. This photocatalytic material's successful MB dye elimination capabilities show its potential role as a leading candidate for wastewater treatment systems. The artificial neural network (ANN) served as the optimization tool for process simulation of degradation. The developed ANN model exhibited exceptional prediction accuracy, which reached a coefficient of determination (R^2) of 0.995

compared to experimental results. This behavior has a major effect on the catalysts' sunlight-driven photocatalytic activity in the present study.

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