Solar Photocatalytic Degradation of Antibiotics in Wastewater by Advanced Oxidation Technology: Optimization using the Response Surface Methodology

Hebatallah Mohammed Khudhair
College of Dentistry, University of Karbala, Karbala, Iraq
hebatallah.k@uokerbala.edu.iq (corresponding author)

Teba Saadi Hussein
Babylon Education Directorate, Ministry of Education, Babylon, Iraq
teba.saadi.ts@gmail.com

Hawraa Jumaa Hashim
Electromechanical Engineering Department, Oil and Gas Equipment Engineering Branch, University of Technology, Baghdad, Iraq
hawraa.j.hashim@uotechnology.edu.iq

Omar Sajer Naser
Department of Medical Laboratory Technologies, Al-Imam University College, Iraq
omarsajer@alimamunc.edu.iq

Maryam Jawad Abdulhasan
Chemical Engineering and Petroleum Industries Department, Al-Mustaqbal University, Babylon, Iraq
mm893505@gmail.com

Received: 29 December 2024 | Revised: 21 January 2025 and 11 February 2025 | Accepted: 15 February 2025

 DOI: https://doi.org/10.48084/etasr.10075

ABSTRACT

Amoxicillin, a widely used antibiotic, is increasingly recognized as an environmental threat due to its persistence in aquatic ecosystems and potential risks to human health. This study investigated the removal of amoxicillin from simulated pharmaceutical wastewater using a solar-powered photocatalytic process with titanium dioxide (TiO_2) in a tube-shaped reactor. The degradation efficiency was assessed by monitoring the reduction in amoxicillin concentration under varying experimental conditions. A Box–Behnken Design (BBD) was applied to evaluate the effects of key parameters, including: initial amoxicillin concentration (10-100 mg/L), TiO_2 dosage (50, 75, and 100 mg/L), hydrogen peroxide (H_2O_2) concentration (200-600 mg/L), and pH levels (3, 5, and 7). The results revealed an optimal degradation efficiency of 90.0% under the following conditions: pH=5, 10 mg/L of amoxicillin concentration, 75 mg/L of TiO_2 dosage, and 400 mg/L of H_2O_2 with a 150-minute exposure to solar irradiation. Statistical analysis using Analysis of Variance (ANOVA) yielded high model accuracy, with R^2 = 96.59%, adjusted R^2 = 93.18%, and predicted R^2 = 81.7%, indicating strong agreement between experimental data and model predictions. The findings confirm the effectiveness of solar-driven photocatalysis in degrading amoxicillin, highlighting its potential as a cost-effective and environmentally sustainable approach for pharmaceutical wastewater treatment.

Keywords-amoxicillin; wastewater; solar photocatalyst; titanium dioxide; advanced oxidation

I. INTRODUCTION

Pharmaceutical products pose a significant environmental threat due to their ability persistence in ecosystems, primarily caused by their resistance to degradation and extremely low biodegradability [1-3]. Antibiotics such as amoxicillin, ciprofloxacin, and tetracycline are widely used to treat bacterial infections worldwide. However, conventional wastewater treatment methods often fail to completely eliminate pharmaceutical residues, necessitating the development of more effective technologies [1,4].

Several techniques have been explored for amoxicillin removal, including filtration, biological treatments, coagulation–flocculation, sedimentation, ozonation, ion exchange, membrane processes, and adsorption. However, managing pharmaceutical wastewater from factories and hospitals—where high concentrations of these contaminants are commonly found—remains a critical challenge for environmental and wastewater engineers globally. Advanced Oxidation Processes (AOPs) have emerged as promising solutions, leveraging hydroxyl radicals (•OH) to degrade both inorganic and complex organic pollutants [5]. Among AOPs, TiO_2 photocatalysis is a particularly effective approach for pharmaceutical wastewater treatment [6]. This technique offers several advantages, including:

- No mass transfer limitations.
- Operation under ambient conditions.
- Cost-effectiveness when utilizing solar irradiation.
- A readily available, inexpensive, non-toxic, and chemically stable catalyst under light exposure [7–9].

Response Surface Methodology (RSM) is a powerful experimental design tool widely used in wastewater treatment optimization. RSM integrates various approaches, such as Face-Centered Composite Design (FCCD), Box–Behnken Design (BBD), and Central Composite Design (CCD), to systematically evaluate process variables [3, 10, 11]. Compared to traditional experimental methods, RSM significantly reduces the number of required experiments while still generating statistically reliable conclusions. It also allows for a comprehensive assessment of variable interactions and their significance. Among these approaches, BBD is particularly efficient, requiring fewer experimental runs while maintaining robust predictive accuracy, making it a widely adopted technique in industrial research [12–15]. This study aimed to investigate the effectiveness of TiO_2 photocatalysis for amoxicillin degradation in simulated pharmaceutical wastewater. The key objectives included:

- Determining the optimal operating conditions—including initial amoxicillin concentration, TiO_2 dosage, pH, reaction time, and H_2O_2 addition—for maximum removal efficiency.
- Utilizing BBD to develop mathematical models describing the degradation process, enabling a detailed evaluation of advanced oxidation mechanisms.
- Validating the proposed model using Analysis of Variance (ANOVA) to assess its accuracy and predictive reliability.

The findings of this study contribute to the advancement of sustainable and cost-effective wastewater treatment technologies, offering a viable solution for mitigating pharmaceutical pollution in aquatic environments.

II. MATERIALS AND METHODS

A. Chemicals

Amoxicillin ($C_{16}H_{19}N_3C_5S$), which has a molecular weight of 365.4 g/mol, was obtained from the Samarra Pharmaceutical State Company. Its molecular structure is shown in Figure 1. The TiO_2-P25 powder (99% purity) used in this study was supplied by Fluka (China), while H_2O_2 (hydrogen peroxide) was sourced from Merck. The pH of the contaminated solution was carefully adjusted using HCl and/or NaOH.

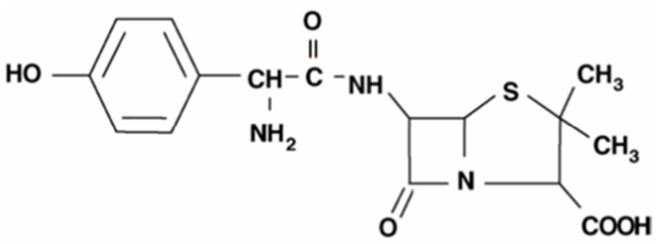

Fig. 1. The molecular structure of amoxicillin.

B. Antibiotics Aqueous Solution

To prepare the antibiotic aqueous solution, a measured quantity of amoxicillin was dissolved in distilled water. This stock solution was prepared weekly and stored at 4°C to maintain stability.

C. Experimental Work and Analysis

The photocatalysis experiments (Figure 2) were conducted outdoors on sunny days between 11:00 AM and 2:00 PM, using a batch reactor model. The reaction was performed in an open 600 mL glass beaker, containing 500 mL of polluted water, surrounded by a mirror reflector to enhance light exposure. A stock solution of amoxicillin antibiotics was prepared by dissolving an appropriate quantity of antibiotics in distilled water. Various concentrations were tested (10, 30, 50, 80, or 100 mg/L) and prepared at different values of pH (3, 5, 7). Subsequently, specific amounts of TiO_2 (50, 75, 100 mg/L) were added. To achieve primary adsorption equilibrium, the mixture was left in the dark for 30 minutes before adding H_2O_2 (200, 400, or 600 mg/L). Sunlight served as the irradiation source. At regular intervals, 10 mL of the sample was collected and centrifuged at 200 rpm for 15 minutes to isolate the catalyst.

D. RSM

To analyze the effects of key variables, RSM was employed, specifically using the Box–Behnken Design (BBD). This approach allowed for the optimization of four factors at three levels, investigating the influence of a) initial amoxicillin concentration, TiO_2 dosage, H_2O_2 concentration and pH level. Table I presents the experimental design, including factor levels and ranges.

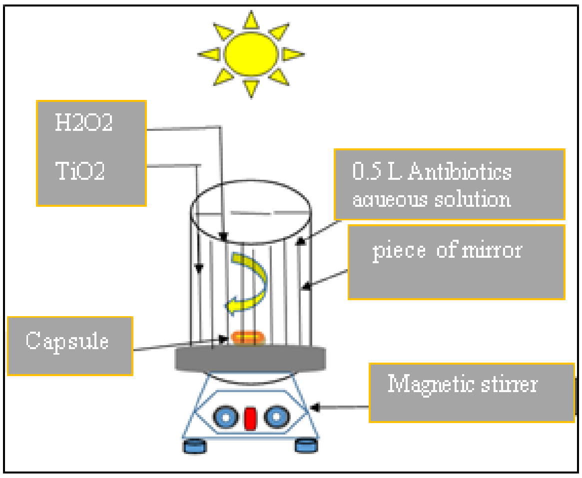

Fig. 2. Diagram of advanced oxidation technology utilizing solar energy.

TABLE I. LEVELS AND RANGE OF EXPERIMENTAL DESIGN

Name	Factors	Range and levels		
		Low level	Medium level	High level
pH	A	3	5	7
Amoxicillin (mg/L)	B	10	50	100
TiO_2 (mg/L)	C	50	75	100
H_2O_2 (mg/L)	D	200	400	600

III. RESULTS AND DISCUSSION

A. Process Optimization

In the optimization process, the variables pH (A), amoxicillin (B), TiO_2 (C), and H_2O_2 (D) were selected to fall within specific ranges, and the responses for amoxicillin removal (R1) were maximized. The optimal process variables were determined to be pH=5, 10 mg/L of amoxicillin, 75 mg/L of TiO_2, and 400 mg/L of H_2O_2, achieving a removal efficiency of 90.0%.

The proposed model is a modified cubic model, which is developed by eliminating larger inconsequential elements to obtain the final empirical model in terms of actual factors (1):

$$R1 = -218.827 + 31.88546 \cdot A + 0.72491 \cdot B + 3.57724 \cdot C + 0.337964 \cdot D - 0.04861 \cdot A \cdot B + 1.03 \cdot 10^{-17} \cdot A \cdot D - 0.00467 \cdot B \cdot C - 0.00056 \cdot B \cdot D - 0.00015 \cdot C \cdot D - 2.76493 \cdot A^2 - 0.00017 \cdot B^2 - 0.02093 \cdot C^2 - 0.00036 \cdot D^2 \quad (1)$$

B. The Removal of Amoxicillin Residue: 3D and 2D Plots

1) Effect of pH on the Photodegradation of Amoxicillin

Figure 3 illustrates the impact of different pH levels (3, 5, and 7) on the photodegradation of amoxicillin. The concentrations of H_2O_2 and TiO_2 were set to 400 mg/L and 75 mg/L, respectively. The key findings were:

- The highest removal efficiency (90%) was achieved at pH 5 after 150 minutes of solar irradiation.
- At pH 3, the removal efficiency was lower compared to pH 5, suggesting that extremely acidic conditions hinder the process.
- TiO_2 exhibits amphoteric behavior, meaning its surface charge changes with pH. The point of zero charge (PZC) of TiO_2 is around pH 6.7. Below this pH, TiO_2 is positively charged; above it, it is negatively charged.
- Electrostatic interactions play a crucial role in amoxicillin degradation. At pH 5, TiO_2 has a higher adsorption capacity and a low amoxicillin concentration, enhancing catalyst-pollutant interaction and facilitating degradation. In acidic environments, the presence of hydrogen ions (H^+) promotes hydroxyl radical (•OH) generation, while dissolved oxygen leads to superoxide radicals ($O_2^{\bullet-}$), both contributing to photodegradation [16–19].

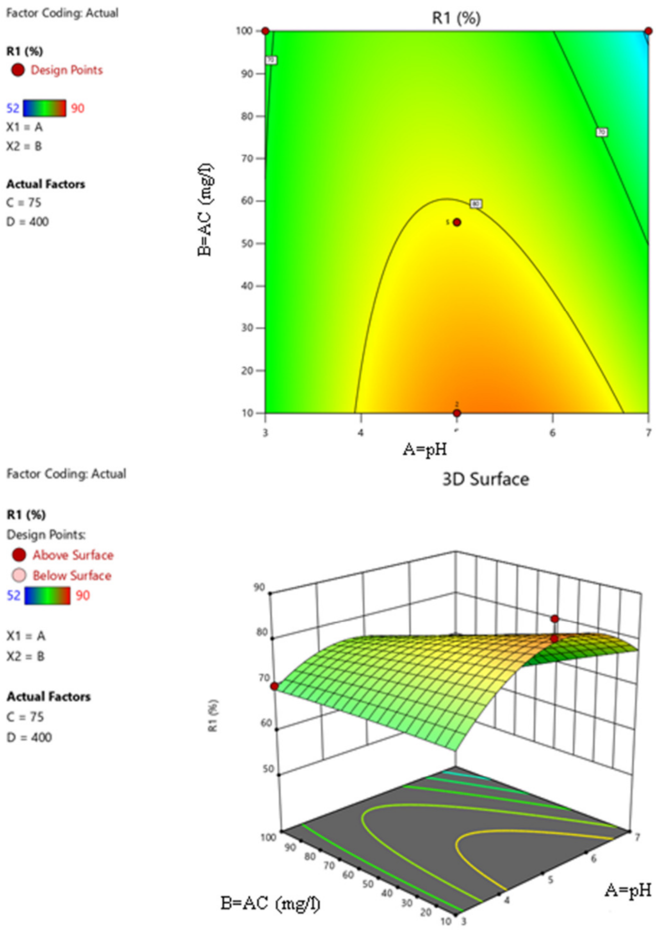

Fig. 3. 2D and 3D surface plot of pH on the photodegradation of amoxicillin.

2) TiO_2 Concentration Effect on the Photodegradation of Amoxicillin

Figure 4 illustrates the impact of different TiO_2 concentrations (50, 75, and 100 mg/L) on the photodegradation of amoxicillin under light exposure. The concentration of H_2O_2 was set to 400 mg/L at a pH of 5. The key findings were:

- Amoxicillin degradation increased with TiO_2 concentration, reaching its peak at 75 mg/L.
- Beyond 100 mg/L, no significant improvement in degradation was observed.

Excess TiO_2 negatively impacted efficiency due to a) reduced light penetration as higher TiO_2 concentrations create a denser suspension, b) increased light scattering, reducing the intensity of light reaching active catalytic sites, and c) agglomeration of TiO_2 particles, decreasing available surface area for photocatalysis [18, 20–22]. These results suggest that optimal TiO_2 concentration is crucial to maximize amoxicillin breakdown without causing light obstruction or catalyst saturation.

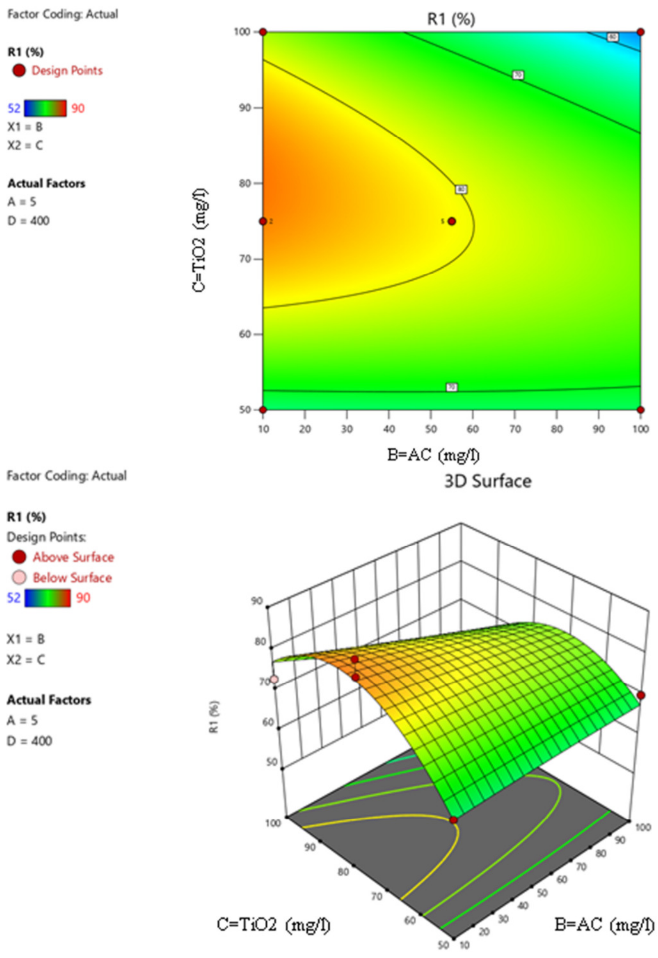

Fig. 4. 2D and 3D surface plot of TiO_2 on the photodegradation of amoxicillin.

3) *The Effect of Initial Concentration of Amoxicillin*

Figure 5 illustrates how varying initial amoxicillin concentrations (10–100 mg/L) impact photocatalytic efficiency. The concentrations of H_2O_2 and TiO_2 were set to 400 mg/L and 75 mg/L, respectively, at a pH of 5. The key finding was that as the initial amoxicillin concentration increased from 10 to 100 mg/L, the removal efficiency gradually declined throughout the photocatalytic process. This decline is attributed to higher adsorption of amoxicillin molecules on the TiO_2 surface, which a) saturates the active catalytic sites, reducing the number of available reactive sites, and b) limits light absorption, decreasing the formation of reactive species essential for photodegradation. These findings align with previous studies, which suggest that higher pollutant concentrations can hinder photocatalysis by blocking active sites on the catalyst surface [23].

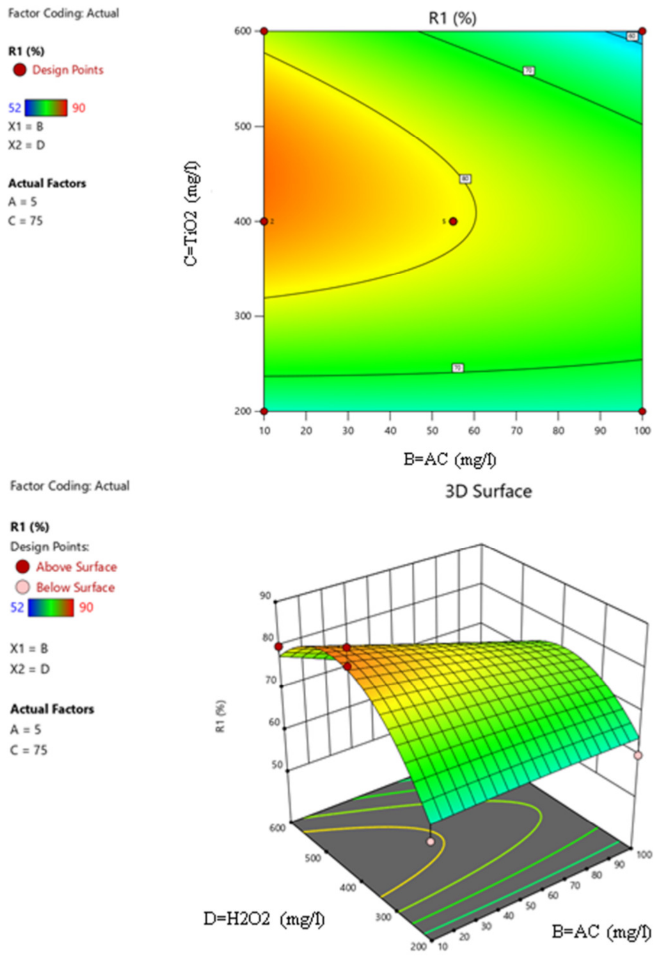

Fig. 5. 2D and 3D surface plot of amoxicillin concentration on the photodegradation of amoxicillin.

4) *H_2O_2 Concentrations*

Figure 6 demonstrates how varying H_2O_2 concentrations (200–600 mg/L) influence the photocatalytic degradation rate of amoxicillin. The concentration of TiO_2 was set to 75 mg/L at a pH of 5. The key findings were:

- For H_2O_2 increasing from 200 to 400 mg/L, the degradation rate initially increased due to the enhanced production of hydroxyl radicals (•OH) via the photo-dissociation of H_2O_2. Hydroxyl radicals act as strong oxidants, accelerating amoxicillin breakdown.

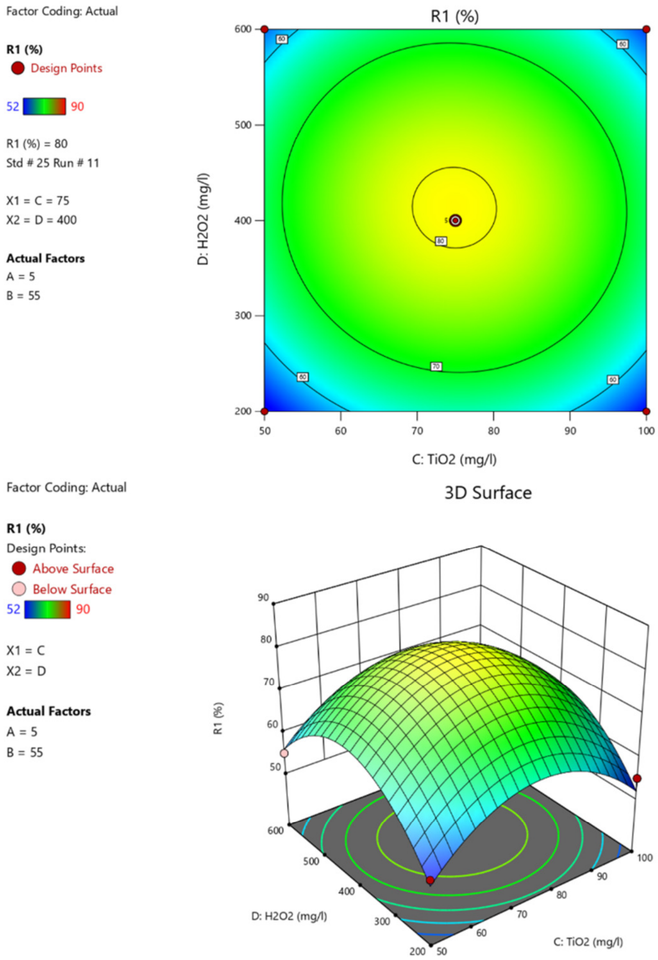

Fig. 6. 2D and 3D surface plot of H_2O_2 on the photodegradation of amoxicillin.

- Beyond 400 mg/L of H_2O_2, the degradation rate declined, likely due to a scavenging effect, where excess H_2O_2 reacts with hydroxyl radicals, reducing their availability for photodegradation.

Authors in [24] found that at higher concentrations, H_2O_2 inhibited catalytic activity by adsorbing onto TiO_2, limiting its active sites. These observations suggest that optimal H_2O_2 concentration is crucial to maximize amoxicillin breakdown.

C. ANOVA

To assess the reliability of the predictive model, ANOVA analysis was performed. Table II presents the results. The Lack of Fit (LoF) analysis showed minimal differences between the residual and pure errors, indicating a good model fit. The correlation coefficient R^2 was calculated at 0.9659, while the adjusted R^2 was calculated at 0.9318, closely aligning with the predicted R^2 at 0.817. The close alignment of these coefficients confirms that a robust polynomial model was successfully fitted to the experimental data [25, 26].

IV. CONCLUSION

This study demonstrated the effectiveness of undoped TiO_2 in the photocatalytic degradation of amoxicillin under optimized conditions. Using Response Surface Methodology (RSM) and Box–Behnken Design (BBD), the influence of key parameters—including pH, amoxicillin concentration, TiO_2 dosage, and H_2O_2 concentration—was systematically evaluated. The optimization process identified the ideal conditions for maximum amoxicillin removal, achieving 90.0% degradation efficiency at pH 5, 75 mg/L of TiO_2, and 400 mg/L of H_2O_2.

TABLE II. ANOVA RESULT FOR AMOXICILLIN REMOVAL

Source	Sum of squares	df	Mean square	F-value	P-value
Model	3791.85	14	270.85	28.31	< 0.0001
A-pH	3.13	1	3.13	0.3266	0.5767
B-TC	272.41	1	272.41	28.47	0.0001
C-TiO_2	0.0833	1	0.0833	0.0087	0.927
D-H_2O_2	44.08	1	44.08	4.61	0.0498
AB	30.63	1	30.63	3.2	0.0952
AC	6.25	1	6.25	0.6533	0.4325
AD	0	1	0	0	1
BC	110.25	1	110.25	11.52	0.0044
BD	100	1	100	10.45	0.006
CD	2.25	1	2.25	0.2352	0.6352
A^2	673.3	1	673.3	70.37	< 0.0001
B^2	0.7042	1	0.7042	0.0736	0.7901
C^2	1143.01	1	1143.01	119.47	< 0.0001
D^2	1371.94	1	1371.94	143.4	< 0.0001
Residual	133.94	14	9.57		
LoF	125.94	9	13.99	8.75	0.014
Pure Error	8	5	1.6		
Cor Total	3925.79	28			

A significant finding of this study is that undoped TiO_2, under optimized conditions, exhibits high photocatalytic activity without requiring additional modifications. While previous research suggests that doping TiO_2 with elements like nickel enhances its photocatalytic properties [27-28], the results of this study indicate that undoped TiO_2 can achieve comparable or even superior efficiency, making it a cost-effective and environmentally sustainable alternative.

Future research should focus on scaling up the process for real-world wastewater treatment applications, assessing its performance in complex water matrices, and investigating degradation pathways and by-products to ensure the environmental safety of treated water. The findings of this study contribute to the development of efficient and sustainable advanced oxidation processes for antibiotic removal from aqueous environments.

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