Photocatalytic degradation of cefoperazone using TiO_2 and ZnO under different irradiation conditions

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Abstract: The increasing environmental concern regarding water pollution has necessitated the development of effective remediation strategies, particularly addressing pharmaceutical contaminants such as cefoperazone. One promising approach involves heterogeneous photocatalysis, a process where semiconductor materials such as TiO_2 and ZnO are employed to facilitate the degradation of organic pollutants under UV or visible light irradiation. In this study, the photolytic and photocatalytic degradation of cefoperazone was investigated under different irradiation types, wherein the applied TiO_2 and ZnO nanomaterials showed significant efficiency in removing cefoperazone compared to direct photolysis. Furthermore, the addition of H_2O_2 enhanced the degradation efficiency of cefoperazone. The dual focus on TiO_2 and ZnO photocatalysts offers a robust solution to mitigate antibiotic pollution in aquatic environments and imposes future trends in environmental sustainability.

Keywords: Photolysis; Photocatalysis; Pharmaceutical analysis; Cefoperazone; Nanomaterials; H_2O_2

1. Introduction

Water is essential for sustaining life on Earth and to tie the Earth's atmosphere, lands, and oceans into an integrated system. The main constituent of the Earth's climate system is the Sun. It emits the solar radiation, which heats the Earth and sets the movement of the natural circulation systems, which affect the environment. In all its forms, water plays a significant and complex role in the climate process and also helps to reflect incoming energy from the Sun to space to keep Earth moderately warm [1]. Global demand for fresh water is increasing constantly with population growth and agricultural development. Also, it is challenging to meet the food demands of a growing population that needs an improved standard of living, which demands clean water supplies to support domestic and industrial

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uses. Even 1.1 billion people do not have access to clean water supplies, while more than 2.6 billion lack access to basic sanitation [2].

Pollution is one of the main concerns of the human population. Contamination and subsequent pollution are mainly brought into the ecosystems by anthropogenic activities. Produced by humans and their activities, contamination has hazardous effects on the environment and resources [3]. Anthropogenic activities lead to the discharge of many pollutants into the environmental waters, which damages the environmental quality and has negative effects on the health of living beings [4]. Water bodies contain many chemical or solid pollutants that are disposed of by anthropogenic contamination, industrial wastes, and leaks. These contaminants are pharmaceutical compounds, pesticides, dyes, detergents, microplastics, heavy metals, etc. What is even more alarming is that these pollutants or their intermediates exhibit toxic effects in the aquatic environments [5].

Nowadays, antibiotics are listed as emerging pollutants due to their continuous input, indiscriminate usage, and persistence in environmental matrices [6]. Cephalosporins are the broad-spectrum β-lactam antibiotics [7]. In Europe, the cephalosporines have reported to be the second most prescribed antibiotic class. Wastewater from cephalosporine production contains active pharmaceutical components, intermediate organic compounds, and inorganic salts, which can exhibit toxic effects and represent a potential threat to the organisms in the environment [8]. Therefore, removal of these antibiotics becomes very important because of the production of toxic effluents and cephalosporin-resistant microbes [9].

Municipal wastewater treatment plants showed as ineffective in completely removing antibiotics and their residues from wastewater. Research endeavors have directed a pathway towards the use of non-conventional treatment methods since they showed great potential in the removal of toxic contaminants from aqueous matrices [10]. Advanced oxidation processes (AOPs) have been shown to be very promising since they surpass the efficiency of conventional oxidation techniques [11]. AOPs can generate reactive oxygen species, such as hydroxyl radical ($^{\bullet}OH$, extremely oxidizing $E° = 2.80$ V, highly reactive, non-corrosive, with a half-life of ~ 1 ns [10]), reacting with organic contaminants through electrophilic reactions, leading to their degradation and often complete mineralization to CO_2, H_2O, and inorganic ions [12].

Taking into account the efficiency of AOPs in the removal of organic pollutants, photodegradation has been shown to be very efficient in pollutant degradation, especially when TiO_2 and ZnO were used as photocatalysts [8]. TiO_2 is widely used because of its high photocatalytic activity, insolubility, chemical and biological stability, nontoxicity, availability, and low price [13]. Application of TiO_2 in heterogeneous photocatalytic oxidation leads to the efficient treatment of wastewater, leading to the obtaining of ultraclean water supplies [14]. However, TiO_2 has certain restrictions regarding its practical

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application. Due to its large band gap, TiO_2 is practically inactive under the sunlight. When TiO_2 is activated by UV radiation, photocatalytic degradation of organic compounds is easily induced [15]. ZnO is also suited for photocatalytic applications owing to its chemical, thermal, and mechanical stability [16]. The band gap energy of ZnO indicates that electrons and holes can be formed with a minimum number of photons. This led to the numerous applications of ZnO. However, the efficiency of ZnO depends on operational conditions, pollutant structure and concentration, radiation, and pH. This often limits its practical applications in large-scale applications and requires further optimization of photocatalytic processes [17].

In this study, we investigated the efficiency of photocatalytic degradation of cefoperazone using TiO_2 and ZnO under simulated solar irradiation (SSI), UV-LED, and UV irradiation. Results were compared with direct photolysis of cefoperazone. Also, the efficiency of cefoperazone photocatalytic degradation by TiO_2 and ZnO was examined with the addition of H_2O_2. TiO_2/H_2O_2 and ZnO/H_2O_2 systems showed high efficiency in the cefoperazone removal compared to the direct photolysis and photocatalysts alone. The kinetics of cefoperazone degradation were monitored by HPLC–DAD (High-Performance Liquid Chromatography with Diode Array Detection).

2. Experimental

2.1. Chemicals and Solutions

Cefoperazone sodium salt ($C_{25}H_{26}N_9NaO_8S_2$, M = 667.65 g/mol, (6R,7R)-7-[[(2R)-2-[(4-ethyl-2,3-dioxopiperazine-1-carbonyl)amino]-2-(4-hydroxyphenyl)acetyl]amino]-3-[(1-methyltetrazol-5-yl)sulfanylmethyl]-8-oxo-5-thia-1-azabicyclo[4.2.0]oct-2-ene-2-carboxylic acid) was used without purification. The standard solution of cefoperazone (0.05 mmol/dm^3) was prepared using ultrapure water (pH = 6.56, κ = 0.055 µS/cm, total organic carbon TOC < LOD). The solution was protected from light and stored at a temperature of 4 °C. The catalysts used were TiO_2 Degussa P25 (75% anatase and 25% rutile-form, specific surface area 50 ± 15 m^2/g, particle size 20 nm) and ZnO (99.9% hexagonal wurtzite structure, particle size around 41 nm, specific surface area 6.5 m^2/g). The other chemicals used as components of the mobile phase for liquid chromatography were 99.9% acetonitrile, C_3H_3NO (Sigma-Aldrich, Massachusetts, United States), and 85% orthophosphoric acid, H_3PO_4 (Lachema, Neratovice, Czech Republic). Also, H_2O_2 (30% Sigma-Aldrich, Massachusetts, United States) was used to enhance radical production and the overall process efficiency.

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2.2. Degradation Procedures

The photocatalytic degradation of cefoperazone was carried out using a Pyrex glass cell equipped with a plain window, onto which the light beam was focused. The total volume of the cell was 40 cm^3. Experiments were performed using 20 cm^3 of 0.05 $mmol/dm^3$ cefoperazone solution containing 1.0 mg/cm^3 of TiO_2 or ZnO, and 3.0 $mmol/dm^3$ of H_2O_2. Before irradiation, the reaction mixture was sonicated for 15 min in the dark to establish adsorption/desorption equilibrium and maintain a constant temperature of 25 °C using a water circulating jacket while exposed to a stream of O_2 at a 3.0 cm^3/min rate. The solution was stirred with a magnetic stirring bar throughout the experiments under a continuous gas flow. A halogen lamp (50 W Philips) was used as SSI source, UV-LED Lamp (5W Enjoydeal, China, type: MR16 AC 85-265V/12) was used as UV-LED irradiation source, and high-pressure mercury lamp (125 W, Philips, HPL-N, emission bands at 290, 293, 296, 304, 314, 335, and 366 nm, with maximum emission at 366 nm) was used as UV irradiation source. All experiments were performed at the natural pH without adjustment.

2.3. Analytical Procedures

To monitor the cefoperazone degradation, HPLC–DAD (Shimadzu) was used. The 0.5 cm^3 aliquots of the solution were taken in selected time intervals. The solutions were filtered through Millipore (Millex-GV, 0.22 μm) membrane filters. The samples were analyzed using an Eclipse XDB-C18 column (150 mm × 4.6 mm, 5 μm particle size, 30 °C), while 10 μL of each sample was injected. The mobile phase consisted of acetonitrile and water, with water acidified using 85% H_3PO_4. Chromatograms were recorded using isocratic elution (ACN : H_3PO_4, 30:70, v/v, pH = 2.42) at a flow rate of 0.8 cm^3/min. The detection was set at the optimal wavelength of absorbance of cefoperazone (λ = 205 nm). The reproducibility of repeated runs was around 3−10%, and the retention time for cefoperazone was determined to be 6.7 $\pm$ 0.1 min. pH of solutions was measured using a combined glass electrode (pH-Electrode SenTix 20, WTW) connected to a pH meter (pH/Cond 340i, WTW).

3. Results and Discussion

3.1. Direct Photolysis

In order to evaluate the influence of different conditions on the efficiency of cefoperazone degradation, the influence of the radiation type (SSI, UV-LED, and UV) at

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natural pH was examined. SSI and UV-LED irradiation proved to be less effective compared to UV irradiation (Figure 1a). The results showed that using SSI and UV-LED irradiation, 2% and 8% of cefoperazone were degraded after 60 min, respectively. With the use of UV irradiation, the degradation efficiency was higher, and after 60 min, 30% of cefoperazone was degraded. The efficiency of this process depends on the absorption spectrum of the investigated compound, and since the absorption maximum of cefoperazone is at 194 nm, the obtained results were expected. During all experiments, the change in pH was monitored at different time intervals. As can be seen from Figure 1b, the application of SSI and UV-LED irradiation did not result in a significant change in pH, it was constant during degradation and was around 7.0. With the application of UV irradiation, the pH decreases from 7.2 to 6.0 after 60 min of irradiation. The change in pH-value from neutral to acidic indicates the formation of acidic intermediates during UV photolysis of cefoperazone.

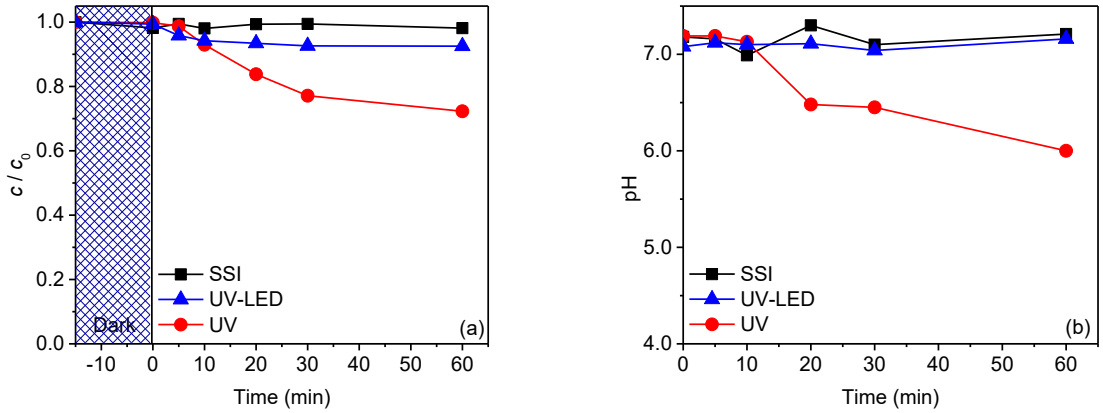

Figure 1. (a) Influence of radiation type on the efficiency of direct photolysis of cefoperazone (0.05 mmol/dm^3) and (b) change in pH.

3.2. Photocatalysis with TiO_2 and ZnO

Since the complete degradation of cefoperazone was not achieved during direct photolysis, heterogeneous photocatalytic degradation was further investigated using commercially available TiO_2 and ZnO photocatalysts using the aforementioned radiation types. First, the efficiency of TiO_2 was investigated (Figure 2). As can be seen, 20% of cefoperazone was adsorbed on the surface of TiO_2 after 15 min (which proved to be sufficient for establishing the adsorption/desorption equilibrium) of sonification in the dark. Compared to direct photolysis, degradation efficiency was significantly higher using TiO_2, and it was similar using SSI and UV-LED irradiation, wherein around 60% of cefoperazone was degraded (Figure 2a). The efficiency of cefoperazone degradation was the highest using UV irradiation, wherein complete degradation of cefoperazone occurred after 60 min. Since

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TiO_2 can absorb radiation of wavelengths up to 400 nm [18], and the absorption maximum of cefoperazone is, as mentioned, located at 194 nm [19], complete degradation of cefoperazone using UV radiation was anticipated. Figure 2b shows the change in pH in different time intervals. Since the kinetics of cefoperazone degradation were similar with the application of SSI and UV-LED irradiation, the change in pH was also very similar, and it decreased from 5.5 to 5.0 after 60 min. Using UV radiation, the pH drops from 5.5 to about 4.0, indicating the formation of more acidic intermediates.

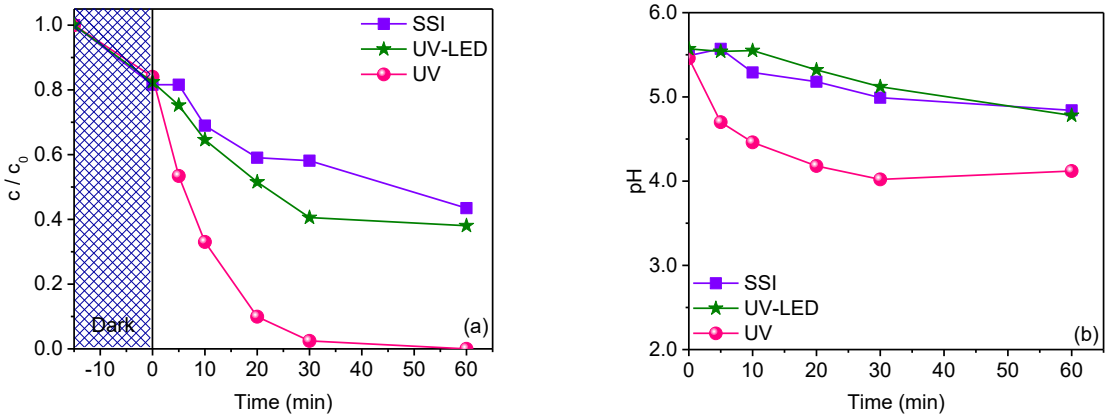

Figure 2. (a) Influence of radiation type on the efficiency of photocatalytic degradation of cefoperazone (0.05 mmol/dm^3) using TiO_2 (1 mg/cm^3) and (b) change in pH.

Further, the effectiveness of ZnO in the degradation of cefoperazone was also tested (Figure 3). Although ZnO and TiO_2 have similar photocatalytic activity, since they have almost the same energy bandgap, they sometimes show different activity in the removal of organic pollutants [20]. As can be seen from Figure 3a, cefoperazone was not absorbed on the surface of ZnO, as in the case of TiO_2 (Figure 2a). Also, it was observed that ZnO exhibited a higher photocatalytic activity in the degradation of cefoperazone compared to TiO_2. Using SSI and UV-LED irradiation, around 70% of cefoperazone was degraded, while in the case of UV irradiation, complete degradation of cefoperazone occurs after 20 min. Figure 3b shows the change in pH value during the degradation of cefoperazone in the presence of ZnO. The pH ranged from 7.0 to 7.5 and did not change significantly during degradation (Figure 3b).

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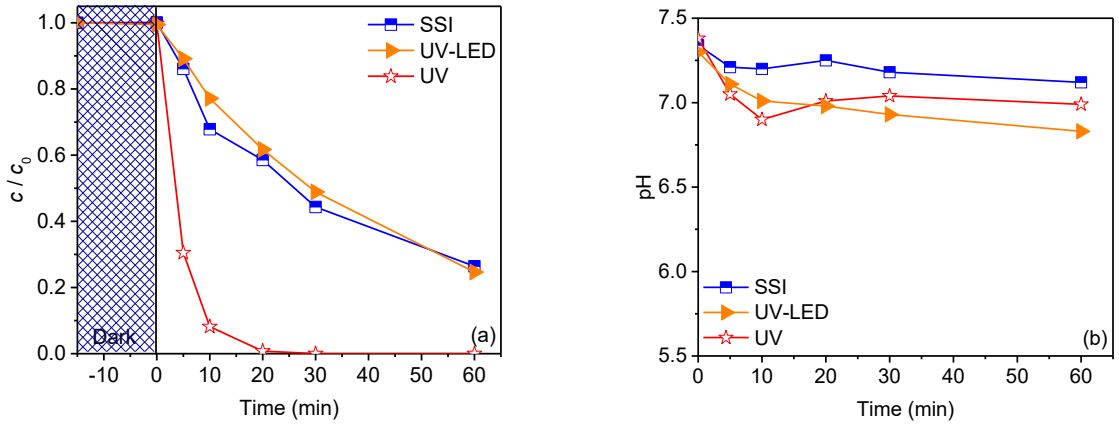

Figure 3. (a) Influence of radiation type on the efficiency of photocatalytic degradation of cefoperazone (0.05 mmol/dm^3) using ZnO (1 mg/cm^3) and (b) change in pH.

3.3. H_2O_2-assisted Photocatalysis

In order to achieve higher photodegradation efficiency of cefoperazone using TiO_2 or ZnO, H_2O_2 was added to the system. In this way, the cefoperazone degradation efficiency increased (Figures 4 and 5) compared to photocatalytic degradation with TiO_2 or ZnO (Figures 2 and 3). The increase in degradation efficiency was most pronounced using UV-LED radiation. After 60 min, cefoperazone degradation efficiency using TiO_2/H_2O_2 under SSI and UV-LED irradiation was 70% and 80%, respectively (vs. 60% using just TiO_2) (Figures 2a and 4a), while using ZnO/H_2O_2 under SSI and UV-LED irradiation, degradation efficiency was 80% and 90%, respectively (vs. 70% using just ZnO) (Figures 3a and 5a). In the presence of UV irradiation, degradation efficiency was similar, since cefoperazone was degraded completely in all cases. Namely, H_2O_2 decomposes into •OH radicals under the influence of irradiation [21]. Therefore, the concentration of •OH radicals was higher with H_2O_2 addition, which resulted in faster cefoperazone degradation. The addition of 3.0 mmol/dm^3 H_2O_2 did not affect the pH (Figures 4b and 5b), and pH was similar to that in solutions where H_2O_2 was not added (Figures 2b and 3b).

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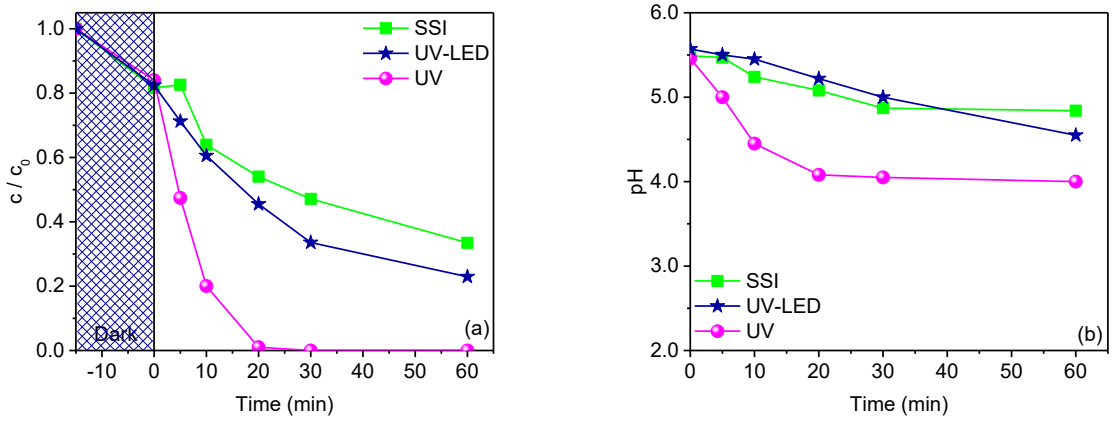

Figure 4. (a) Influence of radiation type on the efficiency of photocatalytic degradation of cefoperazone (0.05 mmol/dm^3) using TiO_2 (1 mg/cm^3) and H_2O_2 (3.0 mmol/dm^3) and (b) change in pH.

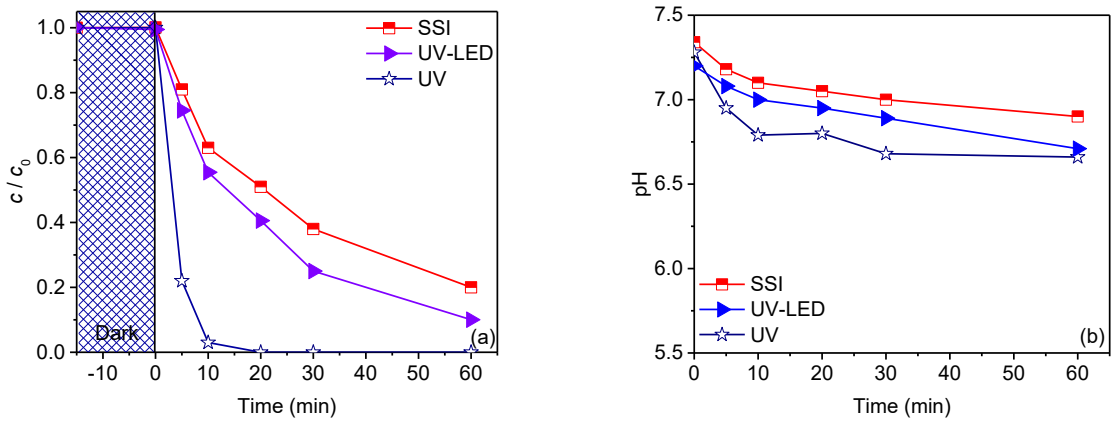

Figure 5. (a) Influence of radiation type on the efficiency of photocatalytic degradation of cefoperazone (0.05 mmol/dm^3) using ZnO (1 mg/cm^3) and H_2O_2 (3.0 mmol/dm^3) and (b) change in pH.

4. Conclusions

In this paper, the efficiency of photocatalytic degradation of cefoperazone using TiO_2 and ZnO photocatalysts and H_2O_2 under different irradiation types (SSI, UV-LED, and UV) was examined. First, the direct photolysis of cefoperazone under SSI, UV-LED, and UV radiation was examined and shown to be inefficient, wherein 2%, 8%, and 30% of cefoperazone were degraded, respectively. Further, the kinetics of photocatalytic degradation using TiO_2 or ZnO under SSI, UV-LED, and UV irradiation were monitored. The applied nanomaterials showed significant efficiency in removing cefoperazone compared to

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direct photolysis. The obtained results demonstrate the effectiveness of heterogeneous photocatalysis over direct photolysis for cefoperazone removal. Among the tested irradiation types, the most effective degradation of cefoperazone was achieved using UV irradiation, wherein complete degradation of cefoperazone was achieved in the case of TiO_2 (after 60 min) and ZnO (after 20 min). ZnO proved to be more efficient in cefoperazone degradation than TiO_2.

Furthermore, the addition of H_2O_2 enhanced the degradation efficiency of cefoperazone and reduced the overall degradation time as a consequence of the increased formation of $^{\bullet}OH$ radicals in the suspension. The most substantial enhancement in process efficiency is particularly evident when employing UV-LED irradiation, wherein, in the case of TiO_2/H_2O_2 80% of cefoperazone was degraded (vs. 60% using just TiO_2). In comparison, in the case of ZnO/H_2O_2 90% of cefoperazone was degraded (vs. 70% using just ZnO). These results show that the combination of semiconductor/H_2O_2 represents an efficient method for the removal of antibiotic contaminants from the environment. The dual focus on TiO_2 and ZnO photocatalysts, at the forefront of advanced oxidation processes, offers a promising approach for mitigating antibiotic pollution and contributes to future sustainable water treatment strategies.

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