

Application of Photocatalytic Process Using UV/TiO₂ for Degradation of Cefepime: A Comparison between Photocatalytic and Photolytic

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ABSTRACT: Cephalosporin is a group of β -lactam antibiotics that has a wide antibacterial ability, so its therapeutic use is high. Although much research has been conducted about the fate of antibiotics in an aquatic environment, little investigation has been done about the removal of cephalosporin. In this study, Cefepime, a fourth-generation cephalosporin antibiotic was selected and different tests such as UV/TiO₂, UV, TiO₂ under darkness, and TiO₂ under sunlight were applied to determine whether these methods are effective ways to remove Cefepime from aqueous solutions or not. Different amounts of catalyst and different pHs were used as effective parameters on degradation efficiency and were optimized. The Cefepime removal was measured by HPLC. The mobile phase was comprised of 10:90 Acetonitrile: water with a flow rate of 1 mL/min. The detected wavelength was 212 nm. Maximum removal efficiency (92.9%) for 20 mg/L Cefepime in neutral pH and catalyst dose of 70 mg/L after 120 min irradiation was observed. At the same conditions, the degradation efficiencies for UV, TiO₂ under sunlight, and TiO₂ under darkness were determined 77.65%, 53.24%, and 15.12% respectively. Also, the reaction rate constant was increased from 0.0054 to 0.0279 (1/min) for photolysis and photocatalysis, respectively. This work was economically compared with photolysis and the result showed that the E_{EO} for the UV/TiO₂ process was five times lower than UV processes. UV/TiO₂ process could be an efficient method to successfully remove the Cefepime antibiotic from aqueous solutions.

KEYWORDS: Cefepime; HPLC; Photolysis; Photocatalysis; Titanium dioxide.

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1021-9986/2021/3/796-803

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INTRODUCTION

Different kinds of pharmaceuticals like antibiotics have been entered into the environment continuously. Although concentration of these compounds is low, they can harm humans and other organisms [1, 2]. Cephalosporin is an important class of antimicrobial agents in both human and veterinary medicine [3]. Cefepime, 7-[α -(2-aminothiazol-4-yl)- α -(z) methoxyiminoacetamido]-3-(1-methylpyrrolidino)-methyl-3-cephem-4-carboxylate is a parenteral fourth-generation cephalosporin that demonstrates good activity against Gram-negative organisms such as *Pseudomonas aeruginosa* and Gram-positive organisms such as *Staphylococcus aureus* [4, 5]. Sewage waste that contains the Cefepime of medicinal plants can cause serious problems in the environment. The efficiency of common treatment processes to remove antibiotic compounds are limited due to the ability of these compounds to persist in the degradation process, high organic load, and toxicity [6, 7]. Different processes have been used to remove antibiotics from the water matrix, such as advanced oxidation processes [8, 9], ion exchange [10], adsorption [11, 12], membrane [13] and biological treatment [14, 15]. Since antibiotics are non-biodegradable, physical and biological treatment systems are not suitable and capable for their degradation them [9]. Advanced oxidation processes (AOPs) are new technologies that many studies have been allocated to in the past few decades [16, 17]. They can treat wastewater that contains recalcitrant organic compounds [6]. AOPs do not transfer pollutants from one phase to another. They also can remove the pharmaceutical pollutants completely and these are the advantages of AOPs over other treatment methods [18]. Titanium dioxide (TiO₂) catalysis has demonstrated ability to decompose a variety of organic pollutants that exist in water and aqueous wastes. The electron/hole pair generates under UV light irradiation with energy more than 3.2 eV. The photo holes (h⁺) from valence band produce OH[·] by adsorbed H₂O that is responsible for complete decomposition of the chemical materials. Additionally, conduction band electrons (e⁻) can react with adsorbed O₂ or other electron accepting species, such as some contaminants [19, 20].

To the best our knowledge, this is the first time that a photocatalytic reaction has been used to illustrate the performance and efficiency of a photocatalytic process

by UV/TiO₂ to degrade the Cefepime antibiotic in an aqueous solution. Hence, the parameters such as solution pH, initial concentration of Cefepime and TiO₂ dosage were studied and the optimal amount of them was obtained. To compare the sample's removal efficiency, the experiment was performed under three different conditions: first, using only UV irradiation; second, using TiO₂ in a dark setting; and third, using TiO₂ under sunlight.

EXPERIMENTAL SECTION

Materials

Cefepime was purchased from Loghman Company (Iran). Acetonitrile and Water both HPLC grade were purchased from Merck. Titanium (IV) oxide for analyzing was purchased from Merck (EMSURE®). All aqueous solutions were prepared in deionized water with an electrical conductivity of 0.05 μ S/cm (Millipore S.A.S 67120 Molsheim). Sodium hydroxide and hydrochloric acid solutions, both 0.2 M, were used to adjust the initial pH of the solutions. All chemicals were in analytic purity and all solvents were used without further purification.

Instrument

Fig. 1 shows the schematics of the photocatalytic reactor that was used in this study. The temperature of this system was fixed at 30 \pm 1 °C by continuous flow of cooling water. As the irradiation source, a medium-pressure UVC lamp (125 W, λ_{max} =247.3 nm) (Shokofan Tosee Company, Iran) was applied and kept into a cylindrical quartz glass to protect the lamp from water. The reactor consisted of a 500 mL beaker. The lamp was placed inside the beaker and illuminated to the solution, containing a specific concentration of Cefepime and a desirable concentration of TiO₂, and stirred with magnetic stirring.

The concentration of Cefepime was determined by HPLC (Shimadzu, Japan). The liquid chromatographic system consisted of Shimadzu 20 AD UFLC with UV-Vis detector and a single pump. Detection was carried out at 212 nm and the Chromatogram was automatically obtained by the LabSolution system software. Injection volume was 20 μ L. The separation was achieved on C18 column (250 mm \times 4.6 mm i.d. and 5 μ m particle size). Mobile phase composition was adopted: 10:90 Acetonitrile: water and flow rate 1 mL/min.

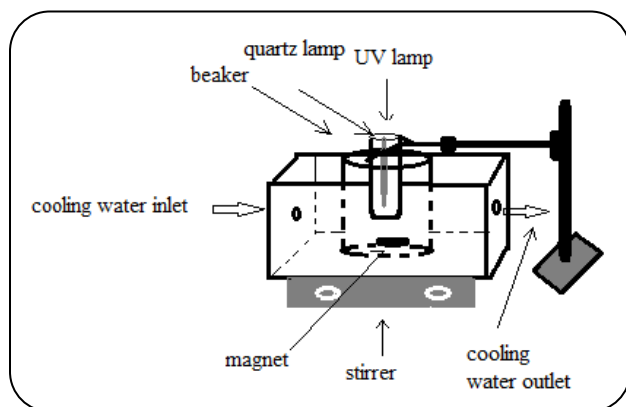


Fig. 1: A schematic of photocatalytic reactor.

Procedures and analysis

A stock solution of 500 mg/L Cefepime was prepared in deionized water and was protected from light at 4 °C. Different working solutions in concentrations of 1, 5, 10, 20 and 50 mg/L were prepared in a volume of 500 mL by diluting stock solution with deionized water.

Then a certain dosage of TiO₂ (1-70 mg/L) was poured into the above solution. Hydrochloric acid or sodium hydroxide was applied to adjust the solution to the desired pH and was measured by the pH meter (Amtast AMT12 (USA)) and then was transferred into the beaker. The mixture was magnetically stirred to ensure thorough mixing during the reaction. The temperature of the photo-degradation system was adjusted by a water bath in which cooling water was recirculated through the jacket of the beaker. During irradiation of the aqueous solution, sample solutions of 0.5 mL were withdrawn from the reactor at specific time intervals. To remove TiO₂ particles, the solution samples were passed through a 0.45 μm membrane filter. Then, the residual Cefepime concentration of the samples was measured by HPLC. Photolysis experiments were performed under optimal conditions that were achieved from the UV/TiO₂ experiment in the absence of the TiO₂ catalyst. For TiO₂ under darkness, a 500 mL beaker containing 20 mg/L Cefepime, 70 mg/L TiO₂ with pH=7 was put in a dark place. The experiment of TiO₂ under sunlight cannot be repeated in two days because the intensity of light may change. Thus, 2 beakers containing 20 mg/L Cefepime, 70 mg/L TiO₂ with pH=7 were put in the same condition. The removal efficiencies of Cefepime for all experiments were calculated by the Equation (1):

$$R = [(C_0 - C) / C_0] \times 100 \quad (1)$$

Where, R is percent removal (%), C₀ and C refer to Cefepime concentration at initial and after the degradation process (mg/L), respectively. A typical HPLC chromatogram of Cefepime is displayed in Fig. 2. All experiments were repeated in duplicate.

RESULTS AND DISCUSSION

Kinetic study

Different studies indicate that Langmuir–Hinshelwood (L–H) is a good kinetic model to describe the kinetics of pharmaceutical photocatalytic degradation in aqueous suspensions [19, 21] (Equation (2)):

$$r = dC / dt = kKC / (1 + KC) \quad (2)$$

Here r is the oxidation rate of Cefepime (mg/L.min), C is the concentration of Cefepime (mg/L), t is irradiation time, k is the reaction rate constant (mg/L.min) and K is the adsorption coefficient of the Cefepime (L/mg). Because concentrations used in this study were in the scale of mg/L, the equation can be simplified to first-order equation as following Eq. (3) [22]:

$$\ln(C_0 / C) = kKt = k't \quad (3)$$

The observed first-order rate constant, k', which is a combination of k and K, could be obtained by plotting ln(C₀/C) versus time.

Effect of catalyst dosage

In order to choose the effective concentration of TiO₂, studies were done in the range of 1-70 mg/L, while the antibiotic dosage and pH were fixed at 20 mg/L and 7, respectively. The removal efficiencies were conducted for specific times in the range of 0-180 min. The results were shown in Fig. 3.

As can be seen, the degradation of Cefepime was almost increased with an increase in the catalyst dosage and the maximum degradation rate was achieved in 70 mg/L TiO₂ concentration. So that by increasing the catalyst dose from 1 mg/L to 70 mg/L, the removal efficiency was achieved from 75.75% to 92.9% after 120 min. With the increase in catalyst dosing, the possibility of access to active sites increases and leads to an increase in photo-degradation efficiency of Cefepime [23]. It is necessary to understand the optimal amount of TiO₂ to avoid extra catalyst [24]. Moreover, the previous studies have stated that after optimal value, degradation efficiency decreases or becomes

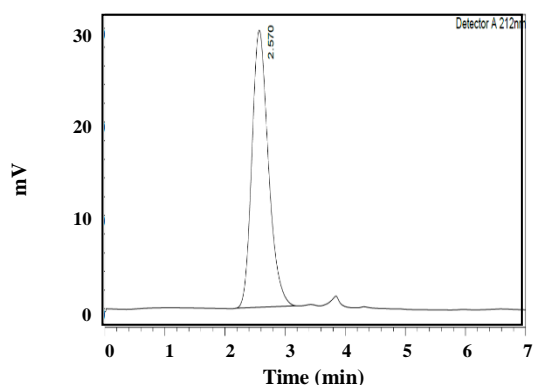


Fig. 2: A typical HPLC chromatogram of Cefepime.

constant, because extreme TiO₂ causes a shadow effect that interferes with transmission of UV light and hinders creation of electron-hole pairs [25].

Effect of pH

To investigate the influence of pH on UV/TiO₂ degradation of Cefepime, experiments were carried out under different pH values varying from 3 to 11 with a constant concentration of catalyst (70 mg/L) and drug (20 mg/L). As can be seen in Fig. 4.a and Fig. 4.b, degradation rate increased with the increasing pH value and at pH 7 reaches its maximum value.

Based on Equation 4, low OH⁻ in acidic mode prevents the formation of hydroxyl radicals and then reduces the degradation rate [26]. Another explanation might be said is that in low pH values, chloride ions can competitively adsorb on the surface of TiO₂ with a big adsorption constant [26].



According to the point of zero charge of TiO₂ (pH_{PZC}=6.8), it can be said that in alkaline conditions, the titanium surface becomes negatively charged [27]. Cefepime has pK_a value of 3.2 [28], thus it is also negatively charged in an alkaline system. Thereby, repulsive force between the catalyst and Cefepime prevents the adsorption of the drug. Moreover, degradation rate increases from 0.0068 to 0.0123 1/min when pH rises from 9 to 11. Because at high pHs, more hydroxide ions are available on the TiO₂ surface and they can be oxidized to the hydroxyl radicals [29]. On the other hand, at alkaline

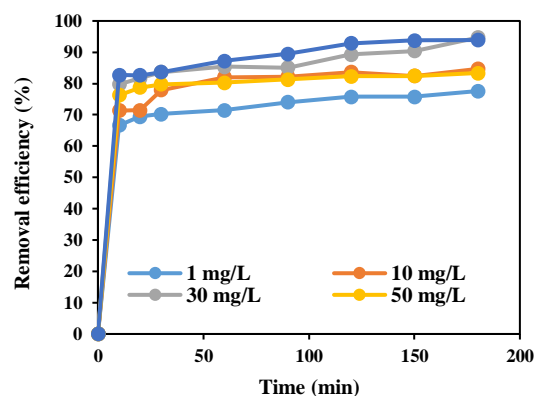


Fig. 3: Effect of TiO₂ catalyst on photocatalytic degradation efficiency (Cefepime concentration= 20 mg/L, pH=7).

condition (above pH_{PZC}), both Cefepime molecules and TiO₂ surface were negatively charged and so repulsive forces between the catalyst and the Cefepime molecules were developed. At pH > pH_{PZC}, the repulsive force between TiO₂ and Cefepime molecules decreased with increasing pH, while the concentration of hydroxyl ions increased at higher pH values. It seems that the higher repulsive force at pH 9, compared to that of pH 7, resulted in a slight decrease in the removal of Cefepime. However, at more alkaline conditions, i.e. pH 11, very high concentration of hydroxyl ions overwhelmed the repulsive force and then improved the removal efficiency at pH 11. The hydroxyl ions, which are available at alkaline condition, can easily be oxidized to form hydroxyl radical as in Reaction (4). Therefore, the removal of Cefepime at pH values of 9 and 11 was mainly controlled by repulsive force and formation of higher hydroxyl radicals, respectively. Overall, the rate of Cefepime removal in UV/TiO₂ system is controlled by two phenomena, i.e. adsorption and degradation by OH radicals. The efficiency of these phenomena changes at various pH values. The experiments showed that the cumulative effect of them reach maximum at pH 7 and then decreased at decreased at pH 9. However, the very high concentration of OH⁻ at pH 11 resulted that the cumulative effect of adsorption and degradation be higher than that of pH 9.

Effect of initial concentration of Cefepime

Different amounts of antibiotics (1-50 mg/L) were considered to assay the influences of initial concentration of Cefepime on the photocatalytic process. As Fig. 5 shows,

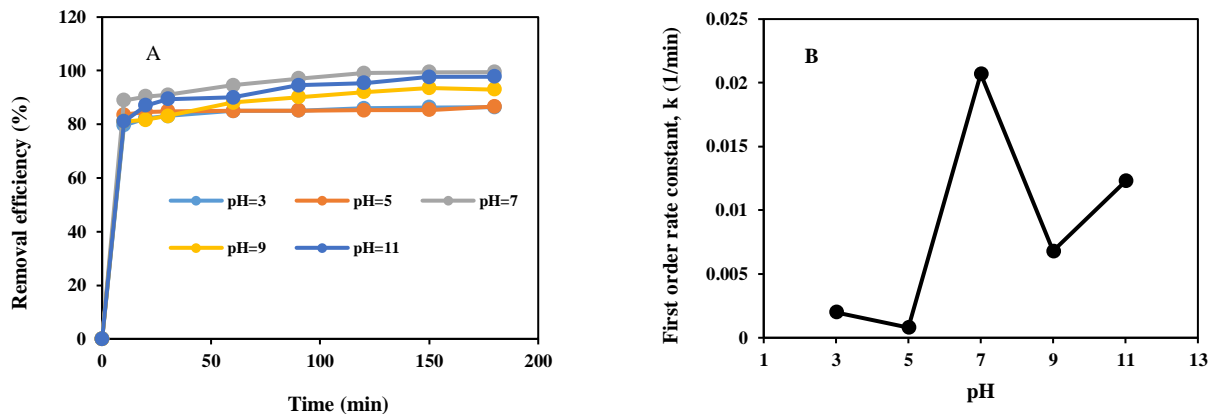


Fig. 4: A) effect of pH on photocatalytic degradation efficiency (Cefepime concentration= 20 mg/L, TiO₂ dosage= 70 mg/L); B) plot of first order constant versus pH during photo catalytic degradation.

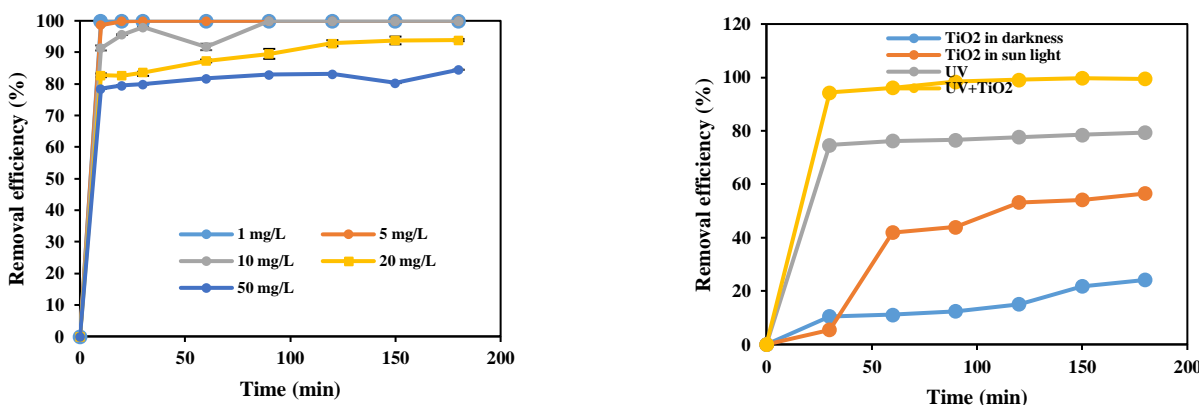


Fig. 5: effect of Cefepime concentration on photocatalytic degradation efficiency (TiO₂ dosage= 70 mg/L, pH=7).

Fig. 6: Effect of different experimental conditions on degradation of Cefepime (Cefepime concentration=20 mg/L, TiO₂ dosage= 70 mg/L, pH=7).

after 30 min irradiation, the degradation efficiency reduced from 100% to 79.9% when the initial concentration increased from 5 to 50 mg/L. High concentration of pollutants prevents penetration of UV light on the catalyst surface and thus fewer hydroxyl radicals are produced and degradation efficiency reduces [30]. One can also say that intermediates and final products produced from the initial Cefepime may compete with Cefepime for adsorption on the catalyst [31].

Comparison

In this study, the experiment was carried out under operating conditions easier than UV/TiO₂ such as UV, TiO₂ under darkness and TiO₂ under sunlight. Our goal was to test different methods and find the best way by taking into account the economic criteria as well as

simplifying the purification systems. For this purpose, under optimal conditions achieved from the UV/TiO₂ experiment, other tests were carried out and the results were reported in Fig. 6.

Removal degradation for UV, TiO₂ under sunlight, and TiO₂ under darkness after 120 min were resulted as 77.7%, 53.2% and 15.1% respectively, but UV/TiO₂ process had been able to remove 92.9% drug concentration successfully at this time. Also, the reaction rate constant was resulted as 0.0054 and 0.0279 (1/min) for photolysis and photocatalysis, respectively. To do an economic comparison between UV/TiO₂ and UV processes due to power consumption, the equations of 3, 5 and 6 of electrical energy per order were used [32]:

$$E_{EO} = (pt1000) / (V 60Ln (C_0 / C)) \tag{5}$$

Table 1: Reaction rate constant calculated by first order equation and E_{EO} determination for the removal of Cefepime (lamp power=125 W, $C_0=20$ mg/L, $TiO_2=70$ mg/L and pH=7).

Process	k' (1/min)	E_{EO} (kWh/m ³)
UV/TiO ₂	0.0279	344.09
UV	0.0054	1777.78

Table 2: A comparison between photocatalytic processes for degrading different kinds of cephalosporin.

Antibiotic	Concentration	Operating conditions	Results and comments	Ref.
Cefazolin	1×10^{-4} mol/L	UVA 365 nm N-doped and undoped TiO ₂ pH=6.4	- No direct photolysis takes place for Cefazolin. - In photo catalytic process by undoped TiO ₂ , 53% of Cefazolin degraded in 60 min. -After 60 min, 80% of Cefazolin was degraded by N-doped TiO ₂ .	(Gurkan et al., 2012)
Cefotaxime	50 mg/L	UV TiO ₂ thin films (64 cm ²) pH=3,7,10	-After 320 min, 96% of Cefotaxime was degraded at pH=7.	(Kondalkar et al., 2014)
Ceftriaxone	10-40 mg/L	hP UV (15W) pH=5-8 TiO ₂ p25 ZnO	-After 120 min, 48.6% of 40 mg/L Ceftriaxone was degraded at pH=6.5. By using ZnO as the catalyst the degradation efficiency was 38.7%.	(Shokri et al., 2016)
Present study	5-50 mg/L	MP UV (125 W) pH=3-11 TiO ₂ =1-70 mg/L	-After 120 min, 77.7% of 20 mg/L Cefepime was degraded by photolysis at pH=7. In the presence of TiO ₂ the removal efficiency reached to 92.9%. -Using Sunlight as the irradiation source could remove 53.2% of the Cefepime.	-

According to the above equations, the following simple equation can be written where P is lamp power (kW), t is time process, V is the volume of irradiated solution (L), C_0 and C are primary and secondary concentration of Cefepime and k' is the first-order rate constant:

$$E_{EO} = (38.4P)/(V k') \quad (6)$$

The results in Table 1 show that the E_{EO} for UV/TiO₂ process is five times lower than the UV process alone, so it can be a good method for wastewater treatment.

Also, a comparison between present work and previous works on the category of cephalosporin were done and the results were compared with this category (Table 2). According to the results obtained from Table 2, a good degradation efficiency and exposure time was achieved over the other works.

CONCLUSIONS

In this study, the use of AOPs to remove the Cefepime antibiotic was investigated. Each of the parameters that affect this process such as TiO₂ dosage, initial concentration of Cefepime, and pH at different times

were evaluated and optimized. The results showed that the rate constant was the maximum at a neutral pH. When the solution containing 5 mg/L Cefepime and 70 mg/L TiO₂ was irradiated for 30 min, complete removal was achieved but by increasing the concentration of Cefepime up to 50 mg/L, degradation efficiency was reduced to 79.9%. Increasing the dose of the catalyst had a positive impact on the process of elimination. According to the results of degradation efficiency and electrical energy per order, the performance of UV/TiO₂ process was five times better than UV alone and also the reaction rate constant of the photocatalysis process was more than five times faster than the photolysis reaction. Eventually, the UV/TiO₂ photocatalytic process may be used to remove the Cefepime antibiotic from aqueous solutions, as a highly efficient technique in a relatively short time.

Acknowledgments

The authors wish to thank the research center at Hormozgan University of Medical Sciences for financial support. Also the authors express their appreciation to the Loghman pharmaceutical.

Received : Nov. 8, 2019 ; Accepted : Feb. 15, 2021

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