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Photodegradation of Antibiotic Using TiO_2 as a Catalyst: A Review

Aprilia Kurnia Asih; Rina Desni Yetti; Boy Chandra
School of Pharmaceutical Science (STIFARM) Padang 25174, Indonesia
E-mail: rinadesniyetti@gmail.com

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Abstract
The use of antibiotics in the community harms the environment. Due to the use of antibiotics is a risk of antibiotic resistance. Photodegradation was a method that could be used to reduce this impact. Photodegradation is a decomposition process using photon energy assistance. The photodegradation process requires a photocatalyst, which is a semiconductor material when subjected to an energy photon. This jump of electrons causes electron holes that can interact with water solvents to form radicals. TiO_2 is the most common photocatalyst that is used for the destruction of pollutants in aqueous solutions. Photochemically, TiO_2 is a stable, non-toxic, widely available, and low-cost manufacturing process. Increasing the concentration of TiO_2 catalyst will also increase the photodegradation activity of antibiotics, and the longer time of the irradiation time will increase the antibiotic degradation.

Keywords: Antibiotics, Photodegradation, TiO_2.

Introduction
Several types of drugs, mainly antibiotics, hormones, preservatives, and anesthetics, have been identified in-ground, waste, and drinking water. The presence of antibiotic waste in water can affect harmful to aquatic organisms as well as causing antibiotic resistance in pathogens in wastewater [1]. Antibiotics are drugs used to treat infections caused by bacteria and other microorganisms. Its toxic properties can inhibit the growth of bacteria (bacteriostatic) or kill bacteria (bactericidal) that contact with these antibiotics [2].

To overcome antibiotic effects on aquatic ecosystems and the environment can be done photodegradation. Photodegradation is a decomposition process (generally organic compounds) with the help of photon energy. This jump of electrons causes electron holes that can interact with water solvents to form radicals [3]. TiO_2 is the most common photocatalyst semiconductor used for the degradation of pollutants in aqueous solutions [4]. Photochemically, TiO_2 is a stable, non-toxic, widely available, and low-cost manufacturing process [5]. To determine the extent of mineralization of the photodegradation process has been done with a TOC analyzer. Total Organic Carbon (TOC) is the number of carbon contained in organic compounds [6]. Classical methods of chemical oxidation for TOC determination are widespread in soil analysis. They are simple and fast, offer high sensitivity [7]. Some studies suggest that the use of TiO_2 catalysts can increase degradation activity. The degradation of thymine over TiO_2 photocatalyst was rapid and significant in aqueous solution under UV irradiation [8]. Combination variation between TiO_2 and activated charcoal (35: 1) for 100 minutes can degrade ciprofloxacin by 82.18% [9]. C-N-S-tridoped TiO2 materials increased the degradation of tetracycline because has a large specific surface area [10].

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Sulfamethoxazole degradation and TOC reduction were improved when TiO_2 concentration was increased, until an optimum located between 0.5–1.0 g TiO_2/L. Degradation 82% of sulfamethoxazole degradation and 23% of TOC reduction [11].

This review focuses on the photodegradation of antibiotic compounds with TiO2 as a catalyst. Also, it can provide some information about the photodegradation using UV-Vis spectrophotometry, High-Performance Liquid Chromatography (HPLC) is used to analyze the degradation of the compound class of antibiotics, and the measurement of Total Organic Carbon (TOC) for mineralization determination from the photodegradation process.

Data Collection

The author created this article review by conducting literature studies. The works of literature were collected from national and international journals in the last twenty years (2010-2020) and pharmaceutical scientific books. The works of literature were collected from trusted online journal sites such as the digital library, ScienceDirect, Elsevier, NCBI, Researchgate, Google Scholar, Springer, and other E-resource with the keyword "photodegradation of antibiotic", "TiO_2 as a catalyst", and "TiO_2".

Analysis Method

The analytical method was used in photodegradation, such as ozonation, ultrasonic waves, Fenton process, and photocatalytic. In the photocatalytic method, TiO_2 was the most commonly used photocatalyst. Photochemically, TiO_2 is a stable, non-toxic, widely available, and low-cost manufacturing process. The visible light absorption of TiO_2 can be influenced by the presence of dopants (Carbon, Nitrogen, and Flour), this is due to a redshift [12]. Photodegradation using TiO_2 catalyst can be measured using UV-Vis spectrophotometer and HPLC as shown in Table 1 and Table 2.

Table 1: Photodegradation measurement of antibiotic compounds using UV-Vis Spectrophotometry

No.	Drug	Concentration	Wavelength	Degradation Results	References
1.	Tetrasiklin	40 mg/L	365 nm	50%	[13]
2.	Ciprofloxacin	0,01 mg/ml	278 nm	88,561%	[14]
3.	Amoxicillin	10 mg/L	387,5 nm	Uv = 25 % Vis = 50%	[15]
4.	Ofloxacin	20 mg/L	288 nm	60%	[16]

To get 50% tetracycline degradation with a concentration of TiO_2 0.5 g/L and tetracycline itself 40 mg/L, 10,20 and 120 minutes irradiation were necessary for UV, solarium, and blacklight lamps, respectively. Tetracycline degradation analyzes were performed in a Shimadzu UV-1603 spectrophotometer as an absorbance measurement, the maximum absorbance at 365nm wavelength [13]. The use of TiO_2-SiO_2 catalyst will increase tetracyclines degradation. The increasing SiO_2 molar ratio will increase tetracycline degradation [17]. Absorbance ciprofloxacin at a concentration of 0.01 mg/ml was measured using Shimadzu UV 1603 spectrophotometer with a wavelength of 278 nm. Determination of ciprofloxacin degradation was conducted at pH 5.8 under a UV lamp at a distance of 5 cm and the addition of 0.05 g of pure TiO_2 nanoparticles, the results obtained 88.561%. No significant degradation when irradiation is not using TiO_2 as a catalyst [14]. Besides, ciprofloxacin can be degraded by TiO_2 and ZnO prepared by sol-gel modified with a pH 10 and 60 minutes, ciprofloxacin degraded about 50% [18]. Exposure

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duration also affected the photocatalytic degradation of ciprofloxacin. for 2, 5, and 30 min Exposure duration, ciprofloxacin degraded were 94%, 97%, and 99%, respectively. This study was conducted by 3.75 mg / L ciprofloxacin concentration and 0.01 g TiO_2 mass [19].

Photocatalytic degradation of Amoxicillin catalyzed by TiO_2 was conducted using a speccord plus UV / Vis spectrophotometer with a wavelength of 387.5 nm. Amoxicillin volume and concentration: 100 mL and 10 mg L-1, photocatalysts mass: 40 mg, time: 24 h, and pH: 6, 25% of amoxicillin degraded by UV and 50% of Vis light [15]. Exposure duration also affected photocatalytic degradation of amoxicillin, for 2, 5, and 30 min Exposure duration, amoxicillin degraded were 94%, 97%, and 99%, respectively. This study was conducted by 10 mg / L ciprofloxacin concentration and 0.01 g TiO_2 mass. Samples of drug solutions were examined using a Perkin Elmer Lambda 16 dual-beam spectrometer with 195 nm wavelength [19].

The photodegradation process of amoxicillin can also be done by the photo-Fenton process with an effective irradiation time of 30 min. However, photodegradation of amoxicillin with photo-Fenton uses a higher cost compared to TiO_2 catalyst [20]. Besides, pH is also very influential in the photodegradation of amoxicillin with photo-Fenton, due to the ionization states of the substrate and the catalyst [21].

The UV / Vis spectrophotometer (Jasco V-530) was used to determine the absorbance of ofloxacin with a wavelength of 288 nm. Analysis of ofloxacin degradation rate by using TiO_2 as the catalyst may degrade ofloxacin by 60%. 0.25-4 g/L TiO_2 concentration was added with ofloxacin 10 mg / L, and pH: 8. The irradiation time required to degrade 60% of ofloxacin is over 120 minutes with an efficient TiO2 concentration of 3 g / L [16].

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Table 2: Measurement photodegradation antibiotic compounds using HPLC (High-Performance Liquid Chromatography)

No.	Samples	Column	Mobile phase	Detector	Chromatographic conditions	Result degradation	References
1.	Amoxicillin	Zorbax Eclipse XDB-C18 column (Agilent)	Water : methanol : acetic acid (200 : 300 : 5)	Detector UV 228 nm	Flow rate: 0,8 cm^3/min vol. injection : 5 µL isocratic elution	60%	[22]
2.	Amoxicillin	L-2130 pump, aL-2200 autosampler, aL-2300 column oven, and an L-2455 DAD	Acetonitrile : methanol: oxalic acid (10:5:80)	Detector UV 231 nm	Flow rate: 0,8 ml/menit vo injection: 20 µL	61%	[23]
3.	Amoxicillin	ZORBAX SB-C18 (4,6 mm × 150 mm, 5 µm) dan suhunya 60 ° C	Water : asetonitril (60 :40)	DAD with λ = 204 nm	Flow rate 0,50 mL / min	58,7%	[5]
4.	Tylosin	BEH C18 reversed phase column (100 mm × 2.1 mm i.d. × 1.7 µm) at 30°C	Acetonitrile: water (20 :80)	Photodiode array detector (AcquityHclass: Waters). λ max (= 232 nm)	Vol injection: 10 µL Flow rate: 0,4 mL/menit	80%	[24]
5.	Spiramycin	BEH C18 reversed phase column (100 mm × 2.1 mm i.d. × 1.7 µm) at 30°C	Acetonitrile: water (20 :80)	Photodiode array detector (AcquityHclass: Waters). λ max (= 232 nm)	Vol injection: 10 µL Flow rate: 0,4 mL/menit	98%	[25]

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Amoxicillin degradation analysis was carried out using HPLC and Zorbax Eclipse XDB-C18 column (Agilent). The analysis was performed by an isocratic method using water: methanol: acetic acid (volume ratio of 200: 300: 5), flow rate 0.8 cm^3 / min and the column temperature was 258 ° C. Injection volume was 5 µL, and UV detection performed at 228 nm. A total of 100 mg/dm^3 amoxicillin concentration and nanocrystal TiO_2 catalyst 2 g/dm^3, increase in amoxicillin degradation rate 60% with the increase of pH [22]. Analysis of amoxicillin concentration of 40 mg / L was carried out using HPLC using acetonitrile (mobile phase A), methanol (mobile phase B), and oxalic acid 0.01 M aqueous solution (mobile phase C) with a ratio (10: 5: 80), an instrument using Hitachi ELITE LaChrom HPLC (Merck-Hitachi, Tokyo, Japan), equipped with L-2130 pump, L-2200 autosampler, L-2300 column oven, and L-2455. Purospher ® RP-18e 125–4 (5 µm; Merck) reverse-phase columns were operated at room temperature (25 ° C). The injection volume was 20 µL and the wavelength of the UV absorbance detector was set at 231 nm. Amoxicillin degraded by 61% with 40 mg/L amoxicillin concentration and 0.5 mg/L TiO2 catalyst [23].

Amoxicillin concentration was determined by High-Performance Liquid Chromatography (HPLC) (Agilent 1100 Series) equipped with a micro vacuum degasser (Agilent 1100 series). Quaternary pump, diode array, and multiple wavelength detector (DAD) (Agilent 1100 series) at 204 nm wavelength. Data recorded by chemistion software. The column was ZORBAX SB-C18 (4.6 mm × 150 mm, 5 µm) and the temperature was 60 ° C. Mobile phase was 60% 0.025 M KH2 PO4 buffer solution in ultrapure water and 40% acetonitrile with a flow rate of 0.50 mL/min. Amoxicillin concentration was 104 mg / L with a pH of 5 for 300 minutes of irradiation and the concentration of TiO_2 catalyst was 2 g / L, amount of degraded amoxicillin by 58.7%. Catalyst concentration is related to the amount of degraded amoxicillin. TiO_2 concentration above 1 g/L does not produce significant antibiotic degradation [5]. It due to reduced light penetration and increased light scattering [26], agglomeration, and high TiO_2 sedimentation below catalyst concentration [27]. With the higher number of TiO_2 concentrations (0-4 mg / L), the photodegradation activity of antibiotics also increases [28].

Experiments of tylosin tartrate photocatalytic degradation were carried out with a batch photoreactor. The UV light used is a Philips PL-S 9W / 10 / 4P lamp. Irradiation was carried out for 300 minutes with an initial concentration of tylosin tartrate 20 mg / L. TiO_2 usage of 1 g/L and initial pH of 4.5. Results obtained 35% and 80% degradation of tylosin tartrate in sequence with UV and UV+TiO_2 radiation [24]. Spiramycin degradation over the initial concentration range of 5–80 mg/L is investigated at constant TiO_2 dose (1 g/L) and natural pH (6.68). It is observed that the time required for the maximum degradation depends on the initial concentration. For the concentrations used in the experiments and at an irradiation time of 300 min, the extent of the Spiramycin removal is 98% [25]. The Spiramycin concentration was monitored by Ultra-High Performance Liquid Chromatography (UHPLC) at λ_{max} (= 232 nm) equipped with a photodiode-array detector. The injection volume and flow rate were respectively 10 µL and 0.4 mL/min. Chromatographic separation was performed with a BEH C18 reversed-phase column (100 mm × 2.1 mm i.d. × 1.7 µm) at 30°C. The mobile phase was 0.1% aqueous solution of formic acid in acetonitrile ACN/ultra-pure water (20 : 80, v/v) [24][25].

Total Organic Carbon (TOC) measurements were used to determine mineralization from the photodegradation process. During the photocatalytic process, the resulted intermediates and products may be more toxic than the parent compound. Therefore, complete mineralization of CO_2 and water is the main target of the treatment process [29]. TOC measurements are shown in table 3.

Table 3: Total Organic Carbon (TOC)

No	Drug	Tool	TOC Result	References
1.	Amoxicillin	Zellweger LabTOC 2100	47%	[22]
2.	Amoxicillin	TNM-1 unit (Shimadzu, model TOC-VCSN)	44%	[23]
3.	Tetracyclines	TOC analyzer (Shimadzu V CHS / CSN , Jepang)	50,4%	[30]
4.	Tetracyclines	Shimadzu 5000 TOC	20%	[13]
5.	Ofloxacin	TOC analyzer (Shimadzu TOC-VCPH/CPN)	50%	[16]

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Measurement of TOC was carried out using Zellweger LabTOC 2100 instrument, with a concentration of 100 mg / dm3 of amoxicillin and 2 g / dm^3 of nanocrystal TiO_2 catalyst. The total organic carbon elimination was 47% after 210 min of irradiation. The amount of amoxicillin degraded was 60%. Indicating that the TOC elimination rate was not proportional to the rate of amoxicillin disappearance [22]. Measurement of TOC analyzed with a TNM-1 unit (Shimadzu, model TOC-VCSN), using amoxicillin concentration of 40 mg/L for 110 min of irradiation, achieving mineralization of 44%. The organic carbon in the solution 21 % is attributed to low molecular weight carboxylate anions, mainly formic, propionic, and maleic acids. However, most of the other organic intermediates still contained sulfur and nitrogen, since only 17 % of the total nitrogen in solution was detected in the form of ammonium [23]. Byproducts that were found on photodegradation antibiotics were nitrogen and sulfur [31].

Measurement of TOC tetracycline at pH 6, with a TiO_2 catalyst 0.4 g / L was determined using a TOC analyzer (Shimadzu VCHS/CSN, Japan) for 180 min. A total 50.4% were achieved for TOC [30]. About 20% of the total organic carbon (TOC) tetracycline was eliminated with an irradiation time of 40 min, complete mineralization can only be obtained with irradiation of 2 hours. TiO_2 concentration used 0.5 g/L using Shimidzu 5000 TOC [2]. TOC of ofloxacin measurement using a TOC analyzer (Shimadzu TOC-VCPH/CPN), with TiO_2 and solar Fenton as a catalyst. The concentration of solar Fenton 5 mg / L was more effective than TiO_2 3 g / L in removing DOC and highest removals of about 50% and 10% in 120 minutes photocatalytic [16].

Conclusion

Based on the description above, TiO_2 is the photocatalyst most commonly used for the destruction of pollutants in aqueous solution. TiO_2 is the most common photocatalyst used for the destruction of pollutants in aqueous solutions. Photochemically, TiO_2 is a stable, non-toxic, widely available, and low-cost manufacturing process. Increasing the concentration of TiO_2 catalyst will also increase the photodegradation activity of antibiotics. High-performance liquid chromatography (HPLC) and UV-Vis spectrophotometry are often used in degradation measurements. Because it provides precise and accurate results, as well as the determination of mineralization from the photodegradation process using TOC (Total Organic Carbon).

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