

Ozone-based Advanced Oxidation Process for pharmaceutical contamination in wastewater: A review

Wahyu Zuli Pratiwi^{1,2*}, Hadiyanto Hadiyanto^{1,2}, and Widayat Widayat^{1,2}

¹Departement of of Chemical Engineering, Diponegoro University, Jl. Prof. Sudarto, SH., Tembalang, Semarang, Central Java, Indonesia

²Center of Biomass and Renewable Energy (C-BIORE), Diponegoro University, Jl. Prof. Soedarto SH, Tembalang, Semarang, 50271, Indonesia

Abstract. Pharmaceutical contaminants, categorized as a subset of Emerging Contaminants, primarily originate from pharmaceutical industries and hospital activities. These pollutants pose significant environmental and public health risks due to their bioactive nature and widespread presence in various ecosystems. Their complex composition in wastewater and low concentrations present a global challenge, as conventional treatment methods often need to be more effective. Advanced oxidation processes (AOPs) have emerged as a promising solution, offering high oxidation efficiency and facilitating the mineralization of such micropollutants. Among various AOPs, ozone-based AOPs stand out because radical hydroxyl is produced with high reactivity. This makes it particularly effective for degrading pharmaceutical micropollutants like antibiotics, which are known for their pseudo-persistent behaviour in the environment. Furthermore, integrating ozone-based AOPs with biological treatments provides a synergistic approach to wastewater treatment, enhancing degradation efficiency while reducing the formation of toxic by-products. This integration can be implemented as a pre-treatment or a post-treatment step, tailoring the process for optimal pollutant removal. This review explores the mechanisms and applications of ozone-based AOPs, focusing on their potential to mitigate pharmaceutical contaminants in wastewater.

1 Introduction

Emerging contaminants (ECs) have become a growing concern over the past decade due to their widespread presence in environmental matrices such as surface water and soil [1]. These pollutants, including nanopollutants and micropollutants, primarily originate from hospital wastewater, industrial discharges, and wastewater treatment plants (WWTPs). Among emerging contaminants (ECs), pharmaceutical contaminants (PCs) are of particular concern due to their bioactive properties, which pose significant risks to human health and environmental ecosystems.

* Corresponding author: wahyuzulip@students.undip.ac.id

Pharmaceuticals, including antibiotics, anti-inflammatory drugs, antidepressants, and lipid-lowering agents, are frequently detected in water sources, with antibiotics and analgesics being the most prevalent [2]. While these compounds serve vital medical purposes, their persistence in the environment at low concentrations (ng/L– μ g/L) poses severe ecological risks [12]. Conventional WWTPs, designed to target easily degradable organic matter, often exhibit removal efficiencies below 10% for persistent pharmaceuticals such as carbamazepine, atenolol, and diclofenac [5]. Moreover, the degradation of parent pharmaceutical compounds does not ensure environmental safety, as their toxic metabolites may persist. Pharmaceuticals enter aquatic ecosystems through various pathways, including direct discharge of wastewater, human and animal excretion, and improper disposal practices. Despite their low concentrations, their accumulation can disrupt aquatic ecosystems, cause synergistic toxic effects, and promote antibiotic resistance in environmental bacterial populations. These factors highlight the urgent need for advanced treatment technologies and comprehensive strategies to mitigate pharmaceutical contaminants' environmental and public health risks [6]. Exposure to sub-inhibitory concentrations of antibiotics below the minimum inhibitory concentration (MIC) can accelerate the emergence of antibiotic resistance genes in microbial communities, altering their structure and function [7].

The concentrations of antibiotics in the environment vary widely, with some being detected at substantial levels. For instance, azithromycin was the most prevalent antibiotic in effluents from WWTPs, with high concentrations identified in influent samples, activated sludge, and sewage [6]. Similarly, non-steroidal anti-inflammatory drugs (NSAIDs) such as diclofenac, ketoprofen, propafenone, and salicylic acid have been detected in groundwater, with concentrations ranging from 91.8 ng/L to 225.2 ng/L [8]. These findings highlight the significant environmental risks of pharmaceutically active substances in aquatic environments. Despite conventional WWTPs, many pharmaceuticals remain unmetabolized and non-biodegradable, leading to their persistent environmental presence. Advanced treatment technologies, particularly AOPs, have emerged as promising solutions for addressing pharmaceutical contaminants. AOPs employ highly reactive hydroxyl radicals (\bullet OH) to oxidize pollutants, transforming them into harmless end products such as CO_2 and inorganic ions. This approach achieves high removal efficiencies and substantially reduces chemical oxygen demand [9,10]. While ozone based AOPs are highly effective in eliminating pharmaceutical pollutants, their implementation in urban wastewater treatment is limited by high operational costs, particularly when targeting complete mineralization, which requires extended treatment durations and significant energy inputs. Despite these challenges, ozonation and AOPs remain among the most efficient and cost-effective methods for treating water contaminated with non-biodegradable pharmaceuticals [11].

Conventional pharmaceutical wastewater treatment primarily relies on biological processes, broadly categorized into aerobic and anaerobic methods [12]. However, these methods often need to be improved for treating large volumes of non-biodegradable and recalcitrant organic pollutants, such as pharmaceuticals, as they are designed to target only biodegradable organic matter. Moreover, pharmaceuticals present in wastewater can disrupt biological processes, further diminishing treatment efficiency. A combination of AOPs and biological treatments has been proposed to overcome these limitations. AOPs can serve as a pre-treatment step to enhance wastewater biodegradability, thereby improving the efficiency of subsequent biological processes by facilitating microbial activity. Alternatively, AOPs can be applied as a post-treatment step to remove residual pollutants after biological treatment.

This review examines the occurrence of micropollutants, particularly antibiotics, in pharmaceutical wastewater and evaluates the application of ozone-based AOPs for their removal. Antibiotics are of significant concern due to their potential for pseudo-persistence in the environment, driven by continuous discharge. Integrating ozone-based AOPs with biological treatments presents a promising strategy for efficiently degrading micropollutants

and generating non-toxic byproducts. This hybrid approach leverages the strengths of both methods: AOPs for their efficacy in breaking down complex pollutants and biological treatments for their cost-effectiveness in managing biodegradable matter.

2 Pharmaceutical contamination in wastewater

Pharmaceutical contaminations in wastewater are primarily generated from various processes in the pharmaceutical industry, including production activities, equipment cleaning, and disposal of off-specification products. This wastewater is characterized by its toxic, recalcitrant nature and contains a mixture of dissolved organic and inorganic compounds. The composition and concentration of pharmaceutical wastewater vary significantly based on the type of product manufactured, production scale, and specific processes involved. According to previous studies [13], pharmaceutical wastewater typically exhibits high pollutant concentrations, with COD values ranging from 2000 to 10,000 mg/L, BOD₅ levels between 1000 and 2500 mg/L, suspended solids at 200–500 mg/L, total nitrogen at 500–1500 mg/L, and total phosphorus at 50–250 mg/L.

This type of wastewater often contains high concentrations of antibiotics, with a complex composition, elevated suspended solids, a low carbon-to-nitrogen ratio, high sulfate content, and significant biological and chromatic toxicity. These characteristics make conventional treatment methods inadequate for complete pollutant removal, leading to the persistence and accumulation of contaminants in the environment [14]. Therefore, advanced treatment technologies are necessary to effectively address pharmaceutical wastewater and minimize its ecological and public health impacts. In this context, wastewater treatment's primary objective is to prevent harm to aquatic ecosystems, safeguard biodiversity, and mitigate risks to fish, shellfish, and other aquatic organisms.

2.1 Antibiotics as pharmaceutical contaminants

Antibiotics are among the most potent agents for treating infections in humans and animals [15]. Medicines that kill bacteria are referred to as bactericidal, while those that inhibit bacterial growth are known as bacteriostatic [9]. Based on their chemical structure, antibiotics are classified into Lactams, Macrolides, Tetracyclines, Aminoglycosides, Quinolones, and Sulfonamides [16,17]. The presence of antibiotics in environments can contribute to the development of antibiotic resistance genes (ARGs) and pose a significant threat to human and environmental health [18]. β -Lactam antibiotics, a class characterized by a lactam ring in their molecular structure, work by inhibiting cell wall biosynthesis in bacterial organisms. These antibiotics are widely used, accounting for approximately 60% of global commercial antibiotic consumption [19]. When released into the environment, β -lactam antibiotics contaminate water bodies, negatively impacting aquatic life. Their persistence can lead to bacterial resistance or immunity to antibiotics. Broad-spectrum β -lactam antibiotics include carbapenems and second-, third-, and fourth-generation cephalosporins, while narrow-spectrum β -lactams include penicillin, first-generation cephalosporins, and monobactams. Macrolides contain a macrocyclic lactone ring and are commonly used to treat respiratory tract and other bacterial infections [20]. Tetracyclines, with linearly fused tetracyclic nuclei, are often found in livestock products, particularly meat. Aminoglycosides consist of two or more amino acids linked by glucosidic bonds to a hexose core and function by inhibiting bacterial protein synthesis [21]. Sulfonamides, used in both human and animal healthcare [22], are bacteriostatic compounds that inhibit organisms' growth by interfering with folate synthesis, which is crucial for purine and pyrimidine nucleotide production. Quinolones are synthetic antibacterial agents used to treat various bacterial infections in humans, derived

from the heterobicyclic aromatic chemical quinoline [23]. Table 1 shows this classification of antibiotics, along with their molecular structure and compound.

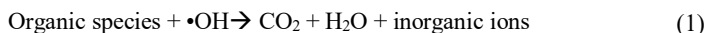
Table 1. Classification of antibiotics, along with their molecular structure and compound.

Antibiotic Class	Compound
β -lactams Penicillins	Penicillin G, cloxacillin, flucloxacillin, dicloxacillin, procaine penicillin G
Carbapenem	Doripenem, Imipenem, Meropenem
Monobactam	Aztreonam
Cephalosporin	1 st generation: Cephalexin, Cefazolin, Cefadroxil, 2 nd generation: Cefoxitin, Cefuroxime, Cefaclor 3 rd generation: Cefoperazone, Cefotaxime, Cefdinir 4 th generation: Cefepime, Cefpirome, Cefzopran. 5 th generation: Ceftaroline, Ceftobiprole, Ceftolozane
Macrolides	Anhydroerythromycin, Clarithromycin, Azithromycin
Tetracyclines	Tetracycline, Demeclocycline, Minocycline, Doxycycline, Tigecycline, Chlortetracycline, Methacycline, Oxy tetracycline,
Aminoglycosides	Amikacin, Paromomycin, Tobramycin, Sisomicin, Isepamicin, Streptomycin, Neomycin, Kanamycin, Spectinomycin, Gentamicin, Netilmicin
Sulfonamides	Sulfamethoxazole, Sulfanilamide, Sulfadiazine, Sulfathalidine, Prontosil, Sulfonamide, Para Aminobenzoic Acid, Sulfisoxazole,
Quinolones	Ofloxacin, Enoxacin, Levofloxacin, Lomefloxacin, Nalidixic Acid, Ciprofloxacin, Norfloxacin, Pefloxacin, Sparfloxacin, Fleroxacin

Antibiotic concentrations in aquatic environments generally remain below 100 ng/L, with some exceptions [24]. For instance, cephalexin and cefradine have been detected at notably high concentrations, reaching up to 1328 mg/L in pharmaceutical production wastewater. The degradation efficiency of antibiotics is influenced by several factors, particularly the initial pH during the treatment process [25]. Ganiyu *et al.* [25] demonstrated the successful degradation of amoxicillin, ofloxacin, cefuroxime, and ciprofloxacin from hospital wastewater, achieving removal efficiencies exceeding 72% for cefuroxime and ciprofloxacin, and more than 92% for amoxicillin and ofloxacin within a reaction time of 15 minutes. Similarly, another study identified the antibiotic meropenem in real pharmaceutical industry waste and reported COD removal efficiencies of 85.4–92.7% using a combination of AOPs and adsorption techniques [26]. Sui *et al.* [27] evaluated large-scale wastewater treatment combining UV and ozonation for the removal of pharmaceutical and personal care products. Their findings revealed that this combined approach effectively eliminated contaminants, with trimethoprim and chloramphenicol showing removal efficiencies exceeding 80%. Due to the typically low concentrations of antibiotics in environmental samples, preconcentration is a critical step before analysis. Liquid chromatography techniques, such as High-Performance Liquid Chromatography (HP-LC) or Liquid Chromatography coupled with Mass Spectrometry (LC-MS), are commonly employed to ensure precise and accurate measurements of antibiotic concentrations in wastewater.

3 Study of removal of pharmaceutical contaminants by Advanced Oxidation Processes

AOPs are innovative and environmentally friendly techniques for pollutant removal [28]. AOPs generate hydroxyl radicals, which are highly reactive and capable of degrading and mineralizing harmful contaminants, transforming them into harmless end products such as CO₂, H₂O, and inorganic salts [29]. The effectiveness of AOPs in enhancing the biodegradability of recalcitrant compounds in wastewater depends on the chemical and physical properties of the target pollutants and the reactivity of the generated free radicals.



AOPs encompass various processes, including chemical and catalytic, mechanical, electrical, photochemical, and photocatalytic methods [30–33]. Many homogeneous AOPs have been utilized, such as ozone, UV/H₂O₂, UV, Photo-Fenton and heterogeneous AOPs such as electro-photo-Fenton, UV/TiO₂, and sunlight-assisted bimetallic catalytic processes to treat wastewater [34]. The AOPs-assisted degradation process encompasses a range of advanced technological methods designed to generate hydroxyl radicals, which act as powerful oxidizing agents. These radicals effectively target and degrade organic contaminants, hazardous wastes, and pollutants in wastewater from industrial, pharmaceutical, hospital, and municipal sources. These processes enhance the suitability of pollutants for bioremediation, reduce pollutant loads, and mitigate toxicity. Combined AOPs demonstrate greater efficiency in addressing global challenges related to environmental waste management [35]. Ozone technology has also been studied in combination. Figure 1. Ozone-based AOPs classification. In this review, chemical and photochemical ozone-based AOPs will be reviewed.

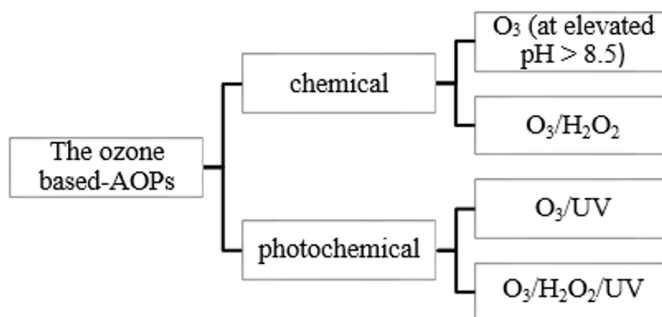


Fig. 1. Classification of ozone-based AOPs.

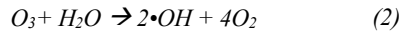
3.1 Basic principles of ozone-based Advanced Oxidation Process

a. O₃ (at elevated pH > 8.5)

Ozone, an allotrope of oxygen also known as activated oxygen, is a highly potent oxidizing agent capable of degrading complex organic pollutants into simpler forms, thereby reducing their toxicity[36]. Ozonation reactions occur via two primary pathways: direct and indirect [37]. The direct pathway involves ozone molecules dissolving in water and reacting with contaminants, while the indirect pathway involves the generation of hydroxyl radicals through ozone decomposition. Ozone is produced by breaking down oxygen molecules into atoms that recombine to form ozone molecules. Due to its strong oxidative properties, ozone can effectively react with many organic pollutants, although not all compounds are

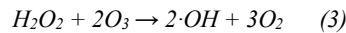
susceptible to direct oxidation by ozone. Conversely, hydroxyl radicals, produced during ozone decomposition, are non-selective and highly reactive, making them integral to AOPs, which are highly effective for eliminating emerging contaminants [38]. Ozone's oxidation potential, approximately 2.07 eV, enables it to disrupt the molecular structure of pollutants. Studies have shown that ozonation performance improves at neutral to alkaline pH levels, as these conditions enhance the decomposition of ozone into radicals, which are primarily responsible for contaminant degradation. Ozone is widely favored for wastewater treatment due to its high reactivity, availability, and cost-effectiveness. Compared to alternative methods such as ultrasound, ozonation demonstrates faster removal rates for many pharmaceutical micropollutants [39]. Ozone is typically generated using an ozone generator and introduced into the reaction system through a reactor.

The efficiency of ozonation in removing micropollutants depends significantly on factors such as pH [27], ozone flow rate [40], temperature [40], and reaction time [41]. Sui et al. [27] highlighted that the removal effectiveness is closely related to the reaction rate constant of the target micropollutant with ozone. Overall, ozonation has shown excellent performance in degrading pharmaceutical micropollutants, making it a promising technology for advanced wastewater treatment applications.



b. O₃/H₂O₂ Process

O₃/H₂O₂ process is a chemically driven electro-peroxide process combining conventional ozonation with the insitu production of H₂O₂ from the electrochemical reduction of oxygen (O₂). Adding hydrogen peroxide and ozone to wastewater accelerates ozone degradation and enhances the generation of hydroxyl radicals [42,43]. Biard et al.[44] investigated the removal of pharmaceutical pollutants, specifically atrazine, from wastewater using a continuous tubular reactor equipped with static mixers. The study revealed that the combination of O₃ and H₂O₂ significantly enhanced the oxidation of atrazine, even at low H₂O₂ concentrations. Furthermore, when peroxide was introduced into the process, the homogeneous tubular reactor demonstrated superior performance compared to a gas-liquid reactor. The radical chain mechanism in this process generates hydroxyl radicals through the reaction of H₂O₂ with ozone [45].



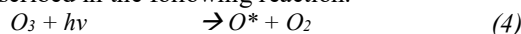
Ozone-based AOPs impact ozone decay kinetics, enhancing its conversion into hydroxyl radicals. Notably, the O₃/H₂O₂ method effectively mitigates bromate formation during the process. However, this approach also results in approximately a 25% increase in ozone consumption [46]. The study shows that the required energy consumption is relatively high.

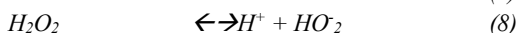
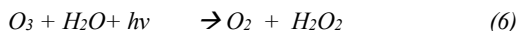
c. O₃/UV

The O₃/UV process utilizes UV photons to activate molecular ozone, resulting in the formation of hydroxyl radicals and H₂O₂, then H₂O₂ breaks down into OH radical points through direct photolysis or dissociation into H⁺ and HO⁻² [47]. Decomposition can occur in three ways:

- (i) via ozonation,
- (ii) by direct UV photolysis,
- (iii) by photolysis of ozone, which produces hydroxyl radicals.

The procedure is described in the following reaction.





O_3/UV has been reported to show high efficiency in treating various organic compounds when compared to UV photolysis or ozonation alone [48]. Applying UV light in ozone mixed water media accelerates the production of reactive OH and improves the performance of the treatment process. Organic molecules are rapidly and wholly degraded when an O_3/UV identification method is used. Many studies have shown the results of the efficiency of O_3/UV treatment in removing various organic and inorganic compounds [49]. As an example of several studies that have been carried out, it is reported that the removal of pharmaceutical substances for sulfamethoxazole micropollutants in wastewater with AOPs using either the O_3/UV method removal can reach 100% [50,51] while with the O_3/H_2O_2 method and the ozonation TOC removal the value is around 90% [46,52]. In the wastewater treatment process, O_3/UV and UV oxidation can be employed with a biological approach to convert complicated contaminants into more biodegradable molecules, allowing more organic compounds to be removed in the subsequent biological process. The process has mild operating conditions, and specific wavelengths of ultraviolet light provide and prevent bromate formation [53].

d. $O_3/H_2O_2/UV$

Lester *et al.* [43] studied the degradation efficiency of the antibiotics trimethoprim, ciprofloxacin and antineoplastic cyclophosphamide in water content in the $O_3/H_2O_2/UV$ process mainly. The evaluation results showed that O_3 contributed more to the degradation of trimethoprim, and O_3/H_2O_2 for ciprofloxacin and UV contributed to a lower level. In $O_3/H_2O_2/UV$ treatment, $\bullet OH$ radicals can be generated from several processes (a) Reaction between O_3 and OH^- , (b) UV photolysis of O_3 , (c) The combined effect of O_3 and H_2O , (d) UV photolysis, of H_2O . The application of $O_3/H_2O_2/UV$ showed a faster degradation time when compared to O_3/UV ; the target pollutant in the form of chlorotetracycline was almost completely degraded within 15 minutes with the $O_3/H_2O_2/UV$ system, and 20 minutes, O_3/UV in the system, but chlorotetracycline did not fully mineralized [43]. In this system, hydrogen peroxide and UV irradiation work synergistically to accelerate ozone decomposition, thereby enhancing the formation rate of hydroxyl radicals. However, the integration of these three components incurs high costs, as such systems are typically restricted to treating wastewater with high organic content and significant microbial populations. Despite the cost, this approach offers the dual benefits of achieving complete mineralization and simultaneous disinfection [43, 54].

3.2 Combination of biological treatment and ozone-based AOPs

The use of biomass, such as activated sludge, fungi, or algae, has been extensively investigated for its potential to treat pharmaceutical wastewater [55–57]. However, due to the complex nature and low biodegradability of pharmaceutical contaminants, non-biological treatment processes are often required to enhance removal efficiency [58]. A combination of biological treatment and ozonation, known as a multistage approach, has been proposed to overcome the limitations of individual methods and leverage their synergistic advantages. This hybrid technology can be implemented in two configurations: ozone-based AOPs as pre-treatment or as post-treatment. Ozone-based AOPs are widely recognized as highly effective in removing recalcitrant pharmaceutical compounds. At the same time, biological

treatments are cost-effective and capable of degrading ozonation by-products into more biodegradable forms.

For example, combining ozonation with aerobic biodegradation has demonstrated remarkable results in pharmaceutical wastewater treatment. Biological treatment achieved a 70% removal of total organic carbon, while ozonation under optimal conditions removed 46%. However, the integrated process of aerobic biodegradation and ozonation achieved 98% TOC removal, with over 99% color removal for amoxicillin-containing wastewater [59]. Furthermore, the combined treatment reduced toxicity by 90%, compared to 62% achieved with ozonation alone. Similarly, Domenjoud *et al.* [60] reported significant improvements in the removal of 14 pharmaceutical compounds, including antibiotics such as sulfamethoxazole and ciprofloxacin, using integrated oxidation and biological processes compared to biological treatment alone.

Pre-ozonation has also been explored for its ability to convert non-biodegradable contaminants into biodegradable forms, thereby facilitating subsequent biological treatment. For instance, Gómez-Pacheco *et al.* [61] demonstrated that pre-ozonation of tetracycline-containing wastewater, followed by activated sludge treatment, effectively degraded tetracycline and mineralized its degradation products. Extended ozonation (up to 120 minutes) significantly reduced chemical oxygen demand (COD) and biological oxygen demand (BOD), highlighting the effectiveness of this combination.

Despite its benefits, pre-ozonation may produce by-products that hinder further biological treatment for certain contaminants, such as those with high amoxicillin concentrations. In such cases, initial biological treatment can target biodegradable fractions, while AOPs address non-biodegradable components. Additionally, biological processes have shown >99% removal of sulfamethoxazole, while ozonation has been particularly effective for recalcitrant compounds like carbamazepine [62]. In conclusion, a systematic multistage approach combining biological treatment and ozone-based AOPs is highly effective for pharmaceutical wastewater treatment. However, the process should be tailored to the specific contaminants present and their potential to form intermediate by-products.

4 Conclusion and perspective

Research on ozone-based AOPs have consistently demonstrated their effectiveness in removing a wide range of pharmaceutical micropollutants, including antibiotics, from wastewater. Pharmaceutical waste is characterized by high organic content and variability in type and concentration, necessitating the implementation of appropriate treatment systems to mitigate its environmental risks. Among various approaches, ozone-based AOPs have proven particularly effective in degrading persistent pharmaceutical contaminants. While biological treatment methods, such as the activated sludge process, are often simpler and more cost-effective, they are less efficient for recalcitrant pollutants.

The integration of AOPs with biological treatments has been explored as a strategy to enhance the remediation of pharmaceutical wastewater. This combined approach is not only more efficient but also environmentally friendly, especially for treating stubborn contaminants. However, AOPs should be reserved for pollutants that resist conventional treatment methods. To optimize these systems, it is crucial to investigate the mechanisms of AOPs and their interactions with different pharmaceutical micropollutants. Furthermore, when scaling up AOPs for large-scale applications, a feasibility assessment of the primary process is essential to ensure operational viability and efficiency.

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