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REVIEW ARTICLE

Advanced oxidation process-mediated removal of pharmaceuticals from water: a review of recent advances

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Abstract

Pharmaceutical compounds have raised significant environmental concerns, due to their persistent and non-biodegradable nature. Addressing their presence in the environment has become a priority, leading to the application of various removal treatment techniques. Advanced oxidation processes (AOPs) undoubtedly have emerged as highly effective removal techniques, as evidenced by the growing body of work in this area. This review offers an overview of the recent advances in the development of AOPs for treating pharmaceuticals and their by-products. Current trends and discoveries reported in diverse AOP studies have been scrutinized and are presented. Furthermore, emphasis is placed on the use of TiO_2-mediated photocatalysis, which stands out as one of the most explored AOPs for pharmaceutical remediation. Performance aspects of TiO_2 photocatalytic treatment are explored and discussed encompassing both commercially available and synthesized TiO_2, as well as engineered TiO_2-based materials (e.g. activated carbon, polymers, metals and non-metals), all aimed at removal of pharmaceutical compounds from the environment. The review concludes by summarizing key findings and offers insights into directions for future research.

Keywords Pharmaceuticals · Transformation products · Advanced oxidation processes · Photocatalysis · Wastewater

Introduction

Water pollution has compelled humans to seek innovative and sustainable technologies, particularly for addressing recalcitrant pollutants, that are resistant to conventional chemical and biological treatments. Pharmaceutical wastewater stands as a significant concern globally, considering the vital role of pharmaceutical compounds or drugs in promoting human and animal health and well-being. As of the end of 2021, the global pharmaceutical market value was estimated to be around USD 1.42 trillion, representing a significant increase compared to 2001, when the market's value was only 390 billion dollars (Mikulic 2022). By 2023, the global pharmaceutical market is anticipated to be worth USD 1.57 trillion. Pharmaceutical manufacturers or industries are obligated to produce highly active and resilient pharmaceuticals to ensure their effective action in humans or animals. The outbreak of COVID-19 is an example of the dependency of humans on drugs, where new anti-COVID-19 drugs were designed to combat the virus. New and existing drugs remdesivir (RDV), dexamethasone (DEX) and hydroxychloroquine (HCQ), despite being effective anti-COVID-19 drugs, have been linked to numerous environmental risks such as the formation of new by-products, during degradation from RDV and the animal toxicity of HCQ (Huang et al. 2022).

Pharmaceuticals from many therapeutic classes are considered micropollutants and contaminants of emerging concern (COECs), due to continuous release from increasing consumption and elimination of human and veterinary medicines. The entry of pharmaceuticals into various waterbodies is through the major gateway, wastewater treatment plants (WWTPs). Other sources of entry include farms, households, industries and hospitals (Fig. 1). The complexity of

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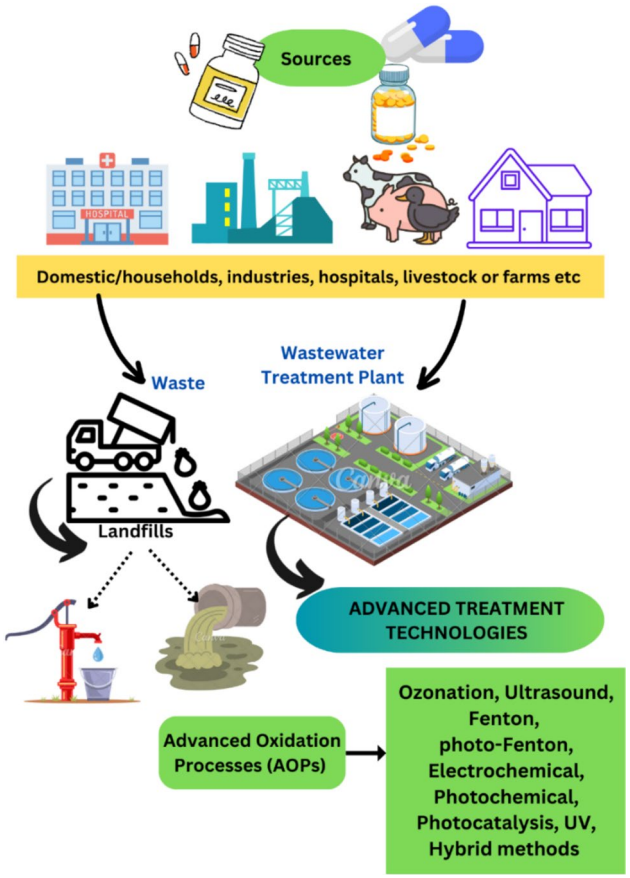

Fig. 1 Entry sources and advanced treatment technologies for pharmaceutical compounds

pharmaceuticals and the simultaneous presence of other pollutants such as personal care products, pesticides and detergents have led to their incomplete removal in WWTPs. Human medications are therefore released into the environment by these WWTPs, while 90% of veterinary medications are excreted in the form of parent chemicals or metabolites through urine and faeces (Graumans et al. 2022; Li et al. 2019). Although the level of pharmaceuticals detected in the environment (e.g. wastewater effluent, river and lake water or sediment) normally ranges from pg/L to µg/L, the levels may also exceed mg/L (Krakko et al. 2022). The presence of these drug residues and metabolites has been associated with significant effects on humans and aquatic life. For example, antibiotic-resistance genes and antibiotic-resistant bacteria are of great concern as pollutants, due to the heavy consumption of antibiotics (Nasrollahi et al. 2022). Antibiotic resistance genes have been detected in pharmaceutical wastewater, urban wastewater and the inlets of WWTPs. Pharmaceuticals in hospital wastewater, which typically range from ng/L to mg/L, could lead to feminization of organisms, and accumulation in various environmental compartments such as soil, plants and animals (Konstas et al. 2019). Detection of pharmaceuticals has been also reported in marine or ocean waters (Madikizela et al. 2020).

Various treatment methods such as adsorption, coagulation, constructed wetland, conventional activated sludge and membrane filtration have been applied for the removal of pharmaceuticals. The removal efficiency generally varies widely between the techniques applied in the WWTPs, the type of WWTPs and types of pharmaceuticals (structure, solubility, concentration) and even country (Eniola et al. 2022; Lozano et al. 2022). These treatment techniques transfer the micropollutants from one phase either to another phase or to an adsorbent, which will eventually generate waste (Graumans et al. 2022). Biological treatment is also not an effective option due to the low biodegradability of pharmaceuticals. As such, advanced oxidation processes (AOPs), which utilize the high reactivity of hydroxyl radicals (HO·) to oxidize organic compounds to innocuous products have been sought for the removal of active pharmaceutical ingredients (APIs), which are contained within pharmaceutical or drug products due to their effectiveness in degrading emerging APIs. Thus far, various types of AOPs have been applied for the removal of APIs which include Fenton and photo-Fenton, ozonation, electrochemical, ultrasonic or sonolysis, photocatalysis and hybrid AOPs (Fig. 1).

The application of AOPs for the removal of pharmaceuticals has remained a hot topic and has attracted the attention of researchers delivering a considerable amount of research. Figure 2 provides an overview of the application of AOPs for the removal of pharmaceuticals, with a particular focus on the researched areas and the targeted Sustainable Development Goals (SDGs). Most of the published work has been centred around SDG 6 clean water and sanitation, signifying the importance attached to addressing issues related to water, sanitation and hygiene. Published reviews on the application of AOPs to the removal of pharmaceuticals are also increasing signifying the importance of addressing the presence of pharmaceutical compounds in our surroundings. For example, a review by Ghazal et al. (2022) summarized various physical processes (membrane and adsorption), nature-based solutions (e.g. constructed wetlands), AOPs and combined processes for the removal of selected pharmaceutically active compounds. Similar topics were also reported in reviews written by Ahmadpour et al. (2024), Kumar et al. (2023) and Liu et al. (2023). Electrochemical-based AOPs (Mosur Nagarajan et al. 2023), photo-induced-based AOPs (Kulišťáková, 2023) and pharmaceutical candidate-based AOP removal (Brillas and Manuel Peralta-Hernández 2023; Feijoo et al. 2023) have also been reported. Majority of these reviews have been based on either types or the targeted pharmaceuticals or certain categories of AOPs, be it single-focused AOPs or those commonly applied, which may not provide a comprehensive understanding of their broader applicability.

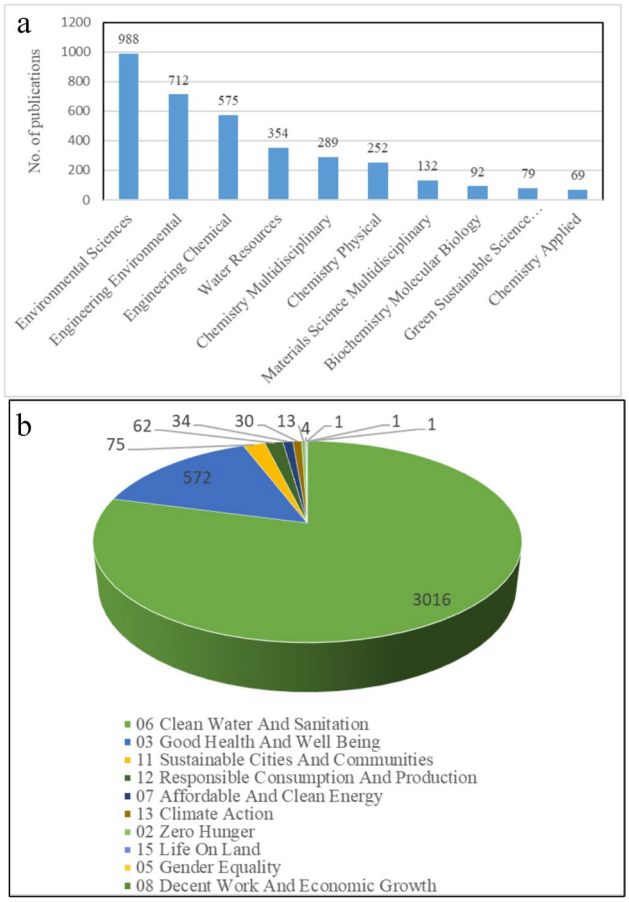

Fig. 2 **a** Correlation between publications and areas of research, **b** number of publications in relation to SDG goals (Web of Science: keywords—Pharma* AND Advanced Oxidation Process)

As such, this review aims to summarize the recent advancements in the application of AOPs for removing pharmaceutical compounds by focusing on various AOPs which include ozonation, Fenton and photo-Fenton, UV and UV/peroxide processes, sonolysis and electrochemical oxidation. The performance of these AOPs related to the removal efficiency of the pharmaceuticals based on their conditions is also discussed. Furthermore, special attention has been given to the application of titanium dioxide (TiO_2) photocatalysis to the removal of pharmaceuticals, particularly focusing on the progress related to both commercial TiO_2 and TiO_2-based nanomaterials. This emphasis is aimed at offering insights into the evolution of TiO_2-mediated pharmaceutical removal, given its significant recognition within the scientific community as an effective approach for eliminating pharmaceuticals from wastewater. This review covers literature from 2018 to the present, building upon our previously published review (Kanakaraju et al. 2018) to provide an up-to-date review of the field.

Pharmaceutical compounds—global trend, occurrence and fate

Active pharmaceutical ingredients

Active APIs can be defined as complex molecules with different functionalities and physicochemical and biological properties. These APIs are mostly polar, and soluble with molecular weights ranging from 200 to 500/1000 Da (Kümmerer 2009; Patel et al. 2019). APIs, which can be either natural or synthetic,have a direct impact on the prevention or treatment of disease, as well as on the restoration, improvement or modification of physiological functioning. All drug products typically contain APIs and excipients (substances other than the API used in drug products, not having an active role in therapeutics but supporting the process to produce an effective product). APIs are classified according to their therapeutic classes such as non-steroidal anti-inflammatory drugs (NSAIDs), antibiotics, beta-blockers, antiepileptics blood lipid-lowering agents, antidepressants, hormones and antihistamines. Although the fate of many pharmaceutical compounds and their metabolites released into the environment remains unknown, three possible fates are as follows: (i) mineralized to carbon dioxide, water and other inorganic ions or acids such nitrate, sulfate and phosphate ions; (ii) retention in the sludge due to lipophilicity and therefore is not readily degradable; and (iii) metabolism to a more hydrophilic form than the parent compound (Halling-Sorensen et al. 1998; Klavarioti et al. 2009; Patel et al. 2019).

North America, Europe, Asia Pacific, Latin America, and the Middle East and Africa make up the regional segments of the global market for APIs (Kenneth Research 2022). North America (USA and Canada) is the world's largest pharmaceutical market with a 49.0% share followed by Europe (23.9%), China (8.2%) and Japan (7.0%) in 2020 (EFPIA 2021). With their extensively developed manufacturing facilities and skilled professionals working in the pharmaceutical industry, China and India have emerged as key producers of APIs. The therapeutic application-based segmentation of the global API market includes communicable diseases, cancer, diabetes, cardiovascular diseases, musculoskeletal disorders, respiratory diseases and other therapeutic applications. Total worldwide consumption data on the APIs used to prevent and treat disease is either scattered or not available mainly due to the practice of unregulated "over the counter" sales of medications (Patel et al. 2019). As such, usage trends and consumption figures are difficult to gather, compute and link. As most pharmaceuticals are not regulated, data storage on pharmaceuticals consumption also varies from country to country. Countries like the USA, Canada, Australia and European

have stringent control measures on pharmaceuticals, while low- or middle-income countries like South Africa struggle with unknown risks of pollution of pharmaceuticals in their environments, due to lack of control measures and monitoring (Hernández-Tenorio et al. 2022).

The employed raw materials, manufacturing processes and the variety of process technologies utilized during the manufacturing of pharmaceutical compounds all have an impact on the pollution rate and wastewater generated during the production of pharmaceuticals. Because of the characteristics of the wastewater produced by pharmaceutical manufacture, it has been designated as a "red category" (Abdelfattah et al. 2022).

Occurrence and fate

Many pharmaceutically active ingredients or APIs have been found in aquatic systems since their first detection in the 1980 s (Mansouri et al. 2021). The fate and behaviour of pharmaceuticals in the environment depend on various factors such as their physicochemical properties, microbial activities and the environmental conditions. Recent studies have shown that pharmaceuticals can undergo various transformation processes such as biodegradation, photodegradation, hydrolysis, oxidation and adsorption (Bavumiragira et al. 2022). The extent and rate of transformation depends on factors such as the pharmaceutical compound's stability, reactivity, persistence in the environment and environmental factors, e.g. sunlight, sediment properties and water chemistry (Bavumiragira et al. 2022; Patel et al. 2019; Pulicharla et al. 2022).

A recent study by Wilkinson et al. (2022) highlighting the compilation of data from different countries to gain a global perspective on the pharmaceutical pollution status has been daunting, due to the wide variety of sample collection techniques and analytical methods applied for detection, and the lack of API detection by some countries. The study highlighted that the regions with the highest total API concentrations were sub-Saharan Africa, south Asia and South America, with caffeine, metformin and carbamazepine the most commonly found APIs. Letsinger et al. (2019) performed a monitoring study with the aim to determine the spatial and temporal occurrence of five APIs in the Humber estuary, England, with ibuprofen detected in the highest concentration. Daily drinking water sampled from the Putrajaya residential areas in Malaysia revealed that the detected APIs may lead to potential human health risks in the long term (Praveena et al. 2019). Table 1 shows the concentrations of pharmaceuticals detected from selected countries.

Unexpected ecological impacts have been caused by pharmaceutical residues. When the pharmaceuticals and their metabolites build up in the environment, these result in unforeseen repercussions. Several studies have supported this: for instance, a monitoring study of pharmaceuticals in seven major rivers in China classified 13 pharmaceuticals as highly toxic, e.g. diclofenac, indomethacin and anhydrous erythromycin, while antibiotics and anti-inflammatory pharmaceuticals were identified as high-risk pharmaceuticals based on their percentage occurrence with 71% and 21% detected, respectively (Li et al. 2019). Zhong et al. (2022) investigated the presence and fate of nine by-products of four antibiotic tetracyclines namely tetracycline, oxytetracycline, chlortetracycline and doxycycline in three WWTPs in Guangzhou, China. The study found that all were detected in the influent, effluent and sludge of the WWTPs. Certain by-products exhibited increased bacterial toxicity, when compared to their corresponding parent compounds.

Amongst numerous pharmaceutical compounds, antibiotics are one of the most frequently detected in various waters of different countries. Antibiotics have been most detected in European countries such as Greece and Spain, where they were found at rates of approximately 6–10%. In Asian nations such as China and Singapore, the frequencies of detection were higher, ranging from 6 to 30% (Omuferen et al. 2022). An investigation of the presence of antibiotics in four rivers in Hanoi, Vietnam, revealed that they were present in highly elevated concentrations ranging from 3050 to 16,700 (median 7800) ng/L which appeared to be much higher than that reported in rivers from other countries (Da Le et al. 2021). Antibiotics were present in the highest concentration compared to other pharmaceuticals in all seven rivers investigated (e.g. SMX 145.29 ng/L in Hai River and 1697 ng/L in Pearl River) (Li et al. 2019).

Studies on environmental risks in terms of ecotoxicological and ecological impact of APIs to aquatic organisms or ecosystems have been gaining importance, but the availability of such data is rather limited. Recent studies have also highlighted the potential risks posed by pharmaceuticals to the environment and human health (Samal et al. 2022; Udebuani et al. 2023). For example, some pharmaceuticals have been shown to cause both acute and chronic effects (Tominaga et al. 2022), cause endocrine disruption in aquatic organisms (Lopez-Velazquez et al. 2023), affect the behaviour of wildlife and contribute to the development of antibiotic resistance in bacteria (Li et al. 2023). Overall, the fate and behaviour of pharmaceuticals in the environment is complex and influenced by various factors, prompting the need for more research to better understand the fate and behaviour of pharmaceuticals in different environmental matrices and to develop effective mitigation strategies to reduce their potential risks to the environment and human health.

Table 1 Concentration of pharmaceuticals in sources of water from selected countries

Country	Analytical technique	Concentration of APIs	Reference
UK (Humber estuary)	Ultra-performance liquid chromatograph coupled with MS/MS (UPLC-MS/MS)	Ibuprofen: 6297.14 ng/L Paracetamol: 916.88 ng/L Diclofenac: 250.8 ng/L Trimethoprim: 247.02 ng/L Citalopram: 42.93 ng/L (Maximum detected concentration)	Letsinger et al. (2019)
Malaysia (Drinking water—residential area of Putrajaya)	Commercial ELISA kits	Caffeine: 0.38 ng/L ± 0.16 (highest) Triclosan: 0.36 ng/L ± 0.16 Ciprofloxacin: 0.32 ng/L ± 0.01 Dexamethasone: 0.32 ng/L ± 0.02 Amoxicillin: 0.31 ng/L ± 0.16 Diclofenac: 0.14 ng/L ± 0.02	Praveena et al. (2019)
Russia (Eastern part of the Gulf of Finland, at coastal and offshore locations)	High-performance liquid chromatography-high-resolution mass spectrometry (HPLC-HRMS)	Diclofenac: 0.9–4.5 ng/L Carbamazepine: 0.7–11.7 Caffeine: 0.6–181.2 Ketoprofen: 1.5–4451.6 Ciprofloxacin: 1.2–6.7 Clarithromycin: 0.6–1.4 Trimethoprim: Not detected (n.d) Drotaverine; n.d Tetracycline: n.d	Chernova et al. (2021)
China (Xiangshan Bay, Zhejiang Province)	UPLC-MS/MS	Carbamazepine: 31 ng/L Lincomycin: 127 ng/L Diltiazem: 0.52 ng/L Propranolol: 1.96 ng/L Venlafaxine: 2.98 ng/L Anhydro erythromycin: 75 ng/L Ofloxacin: 98 ng/L	Sun et al. (2023)
South Africa (Eastern Cape Province—Buffalo, Bloukrans, Swartkops and Tyhume Rivers)	UPLC-ESI–MS/MS and ELISA kit	Carbamazepine: 123.4–36,576.2 ng/L Erythromycin: 11.2–11,800 ng/L Sulfamethoxazole: 20.2–6968 ng/L Clarithromycin: 4.8–3280.4 ng/L Ciprofloxacin: n.d Ibuprofen: n.d (Data of spring season)	Vumazonke et al. (2020)
Ireland (WWTPs)	LC–MS/MS	47 pharmaceuticals were detected Selected pharmaceuticals: Venlafaxine: 8273 ng/L (influent WW) Propranolol: 134 ng/L (surface water) Antipyrine: 1302 ng/L (influent WW)	Rapp-Wright et al. (2023)
Lebanon (Kadicha River and Jeita Spring)	LC–MS/MS	Acetaminophen: n.d–242 ng/L Macrolide: n.d–2806 ng/L Fluoroquinolone: n.d–190 ng/L Carbamazepine: n.d–290 ng/L Diclofenac: n.d–1055 ng/L Carbamazepine: n.d–290 ng/L Sulfonamide: n.d–4100 ng/L	Nassour et al. (2023)

Advanced oxidation processes for the removal of APIs

The wastewater produced by the pharmaceutical industry is often harmful to both aquatic and biological organisms. It has a high chemical oxygen demand (COD) and low biodegradability, which can make it challenging to treat biologically and result in reduced treatment efficiency (Abdelfattah et al. 2022). In this case, AOPs can be an efficient approach for treating pharmaceutical wastewater. The basis of AOPs is centred around the formation of powerful oxidizing or free radical species such as ·OH radicals, ozone, chlorine and permanganate, which effectively break down organic pollutants into less harmful products or molecules.

Among them, the reason for the widespread use of ·OH radical-based AOP techniques in many oxidation technologies is their exceptional oxidation potential (2.8 V), which is higher than most other oxidants, except for fluorine (3.03 V).

Various AOPs have been applied for the removal of APIs in water and wastewater. Amongst them are ozonation, Fenton and photo-Fenton, electrochemical, photocatalysis, photochemical, sonolysis and hybrid processes. The performance of the chosen type of AOPs is governed by various factors such as the type of water, concentration of pharmaceuticals and complexity of water. The application of AOPs for the removal of pharmaceuticals is summarized in Table 2. Figure 3 represents different types of AOPs that have been applied for the removal of pharmaceuticals and key considerations, which impact their efficacy.

Ozonation

Ozonation, a chemical process that involves the use of ozone, a powerful oxidizing agent, to break down and remove contaminants in wastewater (Baghal Asghari et al. 2021), has been identified as an effective method for the treatment of pharmaceutical wastewater. When ozone is added to wastewater, it reacts with organic compounds and other contaminants, breaking them down into simpler, less harmful substances. Ozone can react with organic compounds through either a direct pathway involving molecular ozone, or an indirect pathway involving radicals (Mecha and Chollom 2020). The pH level of the solution also plays a crucial role in determining the major oxidation mechanism during ozonation, as it affects the formation of ·OH radicals. When the pH level is low or under acidic conditions, direct ozone molecule attack becomes the dominant oxidation mechanism. In contrast, when the pH level is high or under basic conditions, ·OH radical attack is the major mechanism (Wang et al. 2020). In alkaline conditions, ozone reacts with hydroxyl ions to generate ·OH radicals, which possess a greater oxidation potential than ozone. Therefore, it is advisable to maintain a pH level close to neutral or alkaline to optimize effectiveness. The complete reaction for generating ·OH radicals with ozone can be expressed as in Eq. 1. Nevertheless, one major drawback of ozone-based processes is the low solubility of ozone in water and the high energy demand for its generation, which results in elevated costs.

$$3O_3 + OH^- + H^+ \rightarrow 2 \cdot OH + 4O_2 \quad (1)$$

Numerous studies have applied ozonation as a tool to treat pharmaceuticals present in water. For example, a study reported that ozonation efficiently removed carbamazepine within 20 min, using ozone dosage of 10 mg/h at pH 7. The mineralization efficiency measured as total organic carbon (TOC) removal was 95%, which appeared to be much better than the another tested method, which is photocatalytic ozonation (TiO_2 and WO_3) (Mathew and Kanmani 2020). A study by Silva et al. (2022) revealed that the degradation of amoxicillin was governed by the ozone concentration (8.13 mg/min and 15.0 mg/min) used at the studied pHs, namely pH 9 and pH 11. In contrast to these studies, the degradation of tetracycline was found to be comparable from pH 3 to pH 11, whereby 99% of removal was achieved after 60 min treatment for all pHs using ultrafine-bubble ozonation (Wang et al. 2020). Nevertheless, TOC removal attained maximum removal of 32% only at pH 11 and TOC was found to decrease with decreasing pH. The fact that 68% of the tetracycline was not converted into CO_2 and H_2O indicates that the mineralization process was not entirely successful. On the contrary, the removal of mixed pharmaceuticals, amoxicillin (AMX), ciprofloxacin (CIP) and acetaminophen (ACET) was 98%, 99% and 98.5%, respectively, via ozonation within 2 min of treatment in the presence of 4 mg/L ozone (optimum concentration) at pH 9, due to the increased production of ·OH radicals at this alkaline pH (Anjali and Shanthakumar 2022). Likewise, the highest mineralization of 89% was also achieved at the same optimum ozone concentration, confirming the mineralization of the mixed compounds into CO_2 and H_2O. Figure 4 shows the proposed degradation pathway of AMX, CIP and ACET via ozonation.

Despite the efficiency of ozonation in treating pharmaceuticals, a recent study by Zilberman et al. (2023) reported that although the ozonation treatment removed iohexol (IHX), bezafibrate (BZF), lamotrigine (LMG), valsartan (VAL), cyclophosphamide (CYP) and sulfamethoxazole (SMX) at levels between 35 and 100% via direct or indirect ozonation (O_3/H_2O_2), mineralization was somewhat low and some of the by-products were found to be more stable and toxic than the parent compounds. SMX was completely removed within 3 min of ozonation producing by-products with aromatic rings and the sulfamide group leading to its stability and non-biodegradability. This finding calls for more advanced technologies to address by-products that are more resistant and toxic than the parent compound.

The current trend shows that the combination of ozonation with other AOPs such as photocatalysis, known as photocatalytic ozonation, is also gaining popularity, due to the synergistic effect generated by semiconductors in the presence of ultraviolet (UV) or visible light and O_3, leading to increased ROS production and thereby facilitating faster and more complete mineralization of pollutants (Orge et al. 2024; Abromaitis et al. 2022; Issaka et al. 2022; Valério et al. 2020). Various catalysts based on metal oxides such as TiO_2, ZnO, MnO_2, WO_3, Fe_2O_3, Fe_3O_4 and others have been combined with ozonation to develop efficient degradation systems. The degradation of sulfonamide antibiotics

Table 2 Summary of the applications of various AOPs in the treatment of pharmaceutical compounds

Types of AOPs applied	Targeted pharmaceuticals	Treatment conditions	Toxicity study	Degradation products identification	Mineralization	Optimized removal conditions or percentage degradation	Reference
Ozonation	Carbamazepine (CBZ)	Ozone flow rate: 5 L/min Ozone dose: 10 mg/h CBZ: 1 mg/L	Not reported	Not reported	TOC: 95% (within 120 min)	Complete CBZ removal within 20 min at pH 7	Mathew and Kanmani (2020)
Ozonation	Amoxicillin	$O_3 + O_2$ rate: 1.0 L/min Ozone doses: 5.00, 8.13, 15.00 and 25.00 mg/min pH: 9–13 AMX: 250 mg AMX/5 mL of simulated WWTP effluent	Not reported	Not reported	TOC: pH 9* (182.3 at 0 min reduced to 177.3 mg/L at 120 min) pH 11*: 196.6 mg/L at 0 min reduced to 195.8 mg/L at 120 min) (*both at 8.13 mg/min O_3)	pH 11 and 15.0 mg/min O_3 produced almost complete degradation of AMX. pH and ozone dosage play important role	Silva et al. (2022)
	Amoxicillin (AMX), acetaminophen (ACET) and ciprofloxacin (CIP)	Ozone flow rate: 2 g/h Ozone dose: 1–5 mg O_3/L	Not reported	LC–ESI–MS	TOC: 89% (at 4 mg/L O_3)	Maximum degradations of the mixture of pollutants AMX (98%), ACET (98.5%) and CIP (99%) were achieved within 2 min with 4 mg/L of ozone	Anjali and Shanthakumar (2022)
Fenton	Amoxicillin	Conc. of $FeSO_4$: 10–40 mg/L Conc. of H_2O_2: 150–600 mg/L pH: 3.0 Amoxicillin: 10 mg/L Ultrapure water	Not reported	Not reported	Not reported	Complete degradation was achieved within 12 ± 2 min (Fe^{2+}: 30 mg/L; H_2O_2: 375 mg/L and pH: 3.0)	Verma and Haritash (2019)
Fenton	Azithromycin	pH: 2.5–4.5 H_2O_2/Fe^{2+}: 8–20 H_2O_2/azithromycin: 120–170 Distilled water	Not reported	Not reported	Not reported	Optimized conditions for 94.13% COD removal were pH = 2.5, H_2O_2/azithromycin = 145 and H_2O_2/Fe^{2+} = 8	Zahi et al. (2023)

Table 2 (continued)

Types of AOPs applied	Targeted pharmaceuticals	Treatment conditions	Toxicity study	Degradation products identification	Mineralization	Optimized removal conditions or percentage degradation	Reference
Photo-Fenton	Carbamazepine	H_2O_2: 0.25–0.75 mg/L Fe^{2+}: 0.05–0.15 mg/L pH: 3.0 Solar simulator (300–800 nm; 1500 W) Ultrapure water	Not reported	GC–MS, NMR	TOC: < 25%	Optimal condition for highest carbamazepine removal (96.4%) was H_2O_2 = 0.75 mg/L, Fe^{2+} = 0.15 mg/L, treatment time = 30 min and irradiance = 450 W/m^2	Parra-Enciso et al. (2023)
Photo-Fenton	Amoxicillin and acetaminophen	Fe^{3+}: 0.5–5.5 mg/L H_2O_2: 0.5–5.0 mg/L pH: 7–8 Solar simulator (2.2-kW xenon arc lamp) Simulated and actual WWTP	Not reported	Not reported	Not reported	Optimal conditions involved Fe(III) concentrations of 3 mg/L, Fe-EDDS ratio of 1:2 and H_2O_2 concentrations of 2.75 mg/L resulted > 90% removal	Hinojosa et al. (2023)
UV photolysis	Metoprolol (MET), atenolol (ATE), bezafibrate (BZB) and atorvastatin	UV lamp (125 W; 200–400 nm) Pure water	Acute toxicity using *P. phosphoreum*	LC–MS	Not reported	Degradation of BZB, ATO, ATE and MET followed pseudo-first-order kinetics with rate constants of 0.1561, 0.1079, 0.0710 and 0.0682 min^{-1}, respectively	Ping et al. (2021)
UV photolysis	Ibuprofen (IBU), salicylic acid (SA), acetylsalicylic acid (ASA) and paracetamol (PAR)	Mercury vapour lamp (250 W; 34.10 mW/cm^2) Ultrapure water	Not reported	Electroscopy ionization with tandem mass spectrometry	Not reported	pH (3–9) controls the photolysis effect on the degradation of IBU, SA, ASA and PAR	Kounaris Fuziki et al. (2023)
UV/H_2O_2	Triclosan and ibuprofen	UVC lamp (254 nm; 36 W) NaOH and MeOH	Not reported	LC–MS	TOC: 10.97% (pH 6 and 15 mg/L H_2O_2 after 75 min)	Degradation of triclosan was 97.69% in 15 min (with 5 mg/L H_2O_2) and ibuprofen was 97.39% after 75 min (with 10 mg/L H_2O_2)	da Luz et al. (2022)

Table 2 (continued)

Types of AOPs applied	Targeted pharmaceuticals	Treatment conditions	Toxicity study	Degradation products identification	Mineralization	Optimized removal conditions or percentage degradation	Reference
UV/H_2O_2	Sulfamethoxazole (SMX), ciprofloxacin (CIP), trimethoprim (TMP), nevirapine (NVP), zidovudine (ZDV) and lamivudine (LMD)	Low-pressure Hg lamp (254 nm; 40 W) Ultrapure water and wastewater effluent	Not reported	Not reported	DOC: 20.3% (in ultrapure water) and 12.1% in wastewater	Removal of TMP, LMD and NVP was 62.9%, 72.2% and 52.9%, respectively. Removal of SMX, CIP and ZDV was also almost ~100% (using 20.4 mg/L H_2O_2 in wastewater)	Ngumba et al. (2020)
Electrochemical	Sulfamethoxazole (SMX), propranolol (PRO) and carbamazepine (CBZ)	Cathode: Ti mesh electrode Anode: Nb/BDD Time: 49.77–100.23 min Current: 0.49–3.01 A Demineralized water and WWTP	Acute toxicity using *Vibrio fischeri*	GC–MS	TOC: 67% (after 120 min)	Optimal conditions for the removal of SMX (86%), PRO (85%) and CBZ (82%) was 90 min of electrolysis and 2.5 A	García-Espinoza and Nacheva (2019)
Electrochemical	Acetaminophen	Electrode: Stainless steel Direct current densities: 12.3 mA/cm^2 (8.5 V), 16.3 mA/cm^2 (10 V) and 20.3 mA/cm^2 (12 V pH: 3–9 Ultrapure water and surface water (dam)	Not reported	HPLC	Not reported	Rate constant of acetaminophen in surface water was much faster (2.5 times more) than in ultrapure water for all pHs and current densities	López Zavala et al. (2020)
Electrochemical	Ciprofloxacin	Electrode: Ni-doped ZnO anode Ti cathode Direct current: 30 V, 3 A UVA and low-pressure lamp Double-distilled water	Daphnia magna	GC–MS	TOC: 83.7%	Photolysis and electrolysis removed only 15% and 34% of the initial 5 mg/L ciprofloxacin, respectively, while photo electrocatalysis achieved a 100% removal rate	Hosseini et al. (2020)

Table 2 (continued)

Types of AOPs applied	Targeted pharmaceuticals	Treatment conditions	Toxicity study	Degradation products identification	Mineralization	Optimized removal conditions or percentage degradation	Reference
Sonolysis	Ranitidine	Frequency: 620 kHz Power: 80 W Ultrapure water	Not reported	LC-TOF/MS	COD: 75%	Sonolysis demonstrated 75% of COD removal and the degradation of ranitidine followed pseudo-first-order kinetics with rate constant of 0.071 min^{-1}	Elias et al. (2019)
Sonolysis	Paracetamol	Frequency: 22–2000 kHz Milli-Q water	Not reported	Not reported	Not reported	Rate constant demonstrates a linear increase with rising frequency within the range of 200 to 850 kHz, particularly notable in the case of dual frequency	Zare et al. (2023)
Sonolysis	Acetaminophen, cloxacillin, diclofenac, naproxen, piroxicam, sulfacetamide (SAM) and cefadroxil (CDX	Frequency: 40–1175 kHz Acoustic power: 1–24.4 W Distilled water and mineral water	Not reported	Not reported	Not reported	Degradation of all pharmaceuticals was compound dependent and affected by water matrix	Camargo-Perea et al. (2021)

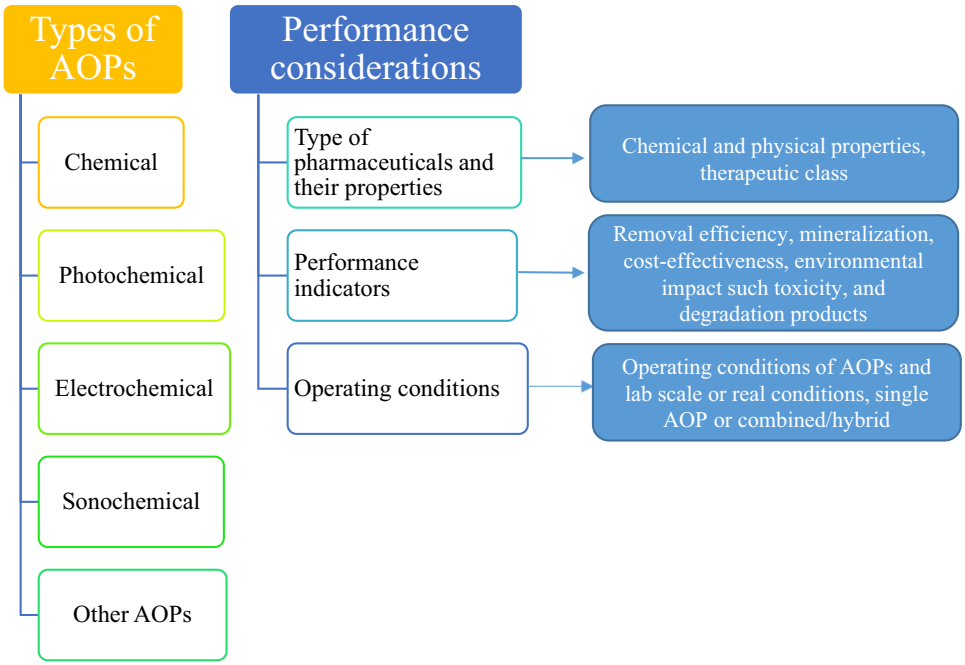

Fig. 3 Classifications and key consideration of AOPs for pharmaceuticals removal

namely sulfacetamide, sulfathiazole, sulfamethoxazole and sulfadiazine were investigated using TiO_2/O_3 and TiO_2/UV/O_3 systems by Esrafili et al. (2020), while Yang et al. (2018) explored to the role of WO_3/Visible/O_3 in the degradation of cephalexin. Both TiO_2/UV/O_3 and WO_3/Visible/O_3 were efficient in degrading the APIs, when compared to ozonation only and catalytic ozonation. Figueredo et al. (2019) compared TiO_2 P25 and WO_3 for the removal of primidone. Photocatalytic ozonation mediated by synthesized TiO_2 nanotube arrays showed higher removal for ciprofloxacin ($0.041 \pm 0.42\ min^{-1}$) compared to photocatalysis only ($0.007 \pm 0.02\ min^{-1}$) (Abromaitis et al. 2022). The performance was observed to be controlled by the TiO_2 properties and conditions applied during the treatment.

Pre-treatment by ozonation and followed by biological treatment demonstrated high removal of COD for synthetic wastewater containing ciprofloxacin and real pharmaceutical wastewater with the removal of 68% and 82.5%, respectively, and concluded that synergistic effects of these treatments were efficient in treating less biodegradable pharmaceutical wastewater (Mohan and Balakrishnan 2021). Although studies have demonstrated that catalytic ozonation may be efficient in removing pharmaceuticals, by-products associated with this treatment continue to present a challenge (Issaka et al. 2022; Zilberman et al. 2023). A recent study explored the combination of UV and O_3 to address the limitations of ozonation, which can be costly and often results in incomplete mineralization of organic pollutants due to complex degradation pathways (Kim et al. 2024). The combined 1.5 mg/L O_3/UV treatment produced > 97% of removal of contaminants in synthetic and hospital wastewaters within 7 min due to the production of ·OH radicals and the degradation products also displayed less toxicity based on in-silico toxicity predictions.

Ozonation is a promising AOP for pharmaceutical wastewater treatment, offering rapid degradation of various APIs and flexibility across different pH conditions. It can enhance mineralization, especially when combined with photocatalysis or biological treatment, and has demonstrated high removal efficiencies for pharmaceuticals like carbamazepine and ciprofloxacin. However, there are still some challenges associated with ozonation. These include the formation of potentially toxic by-products and the high energy consumption requirements. Moreover, the effectiveness of ozonation can be influenced by a variety of factors, including the specific contaminants present in the wastewater, the ozone dose and contact time, and the pH and temperature of the wastewater. Therefore, the optimization of ozonation processes for pharmaceutical-laden wastewater requires consideration and evaluation.

Fenton and photo-Fenton

Fenton and photo-Fenton processes have been proven to be effective in remediating wastewater containing pharmaceutical compounds. The Fenton reaction, which was discovered in 1894 by H.J.H. Fenton, has been applied to remove PACs from water along with Fenton in the presence of light (photo-Fenton). The ·OH radicals formed during the Fenton reaction are responsible for the degradation of PACs to small molecules (Eqs. 2–4). The homogeneous Fenton reaction is thought to have certain unavoidable downsides,

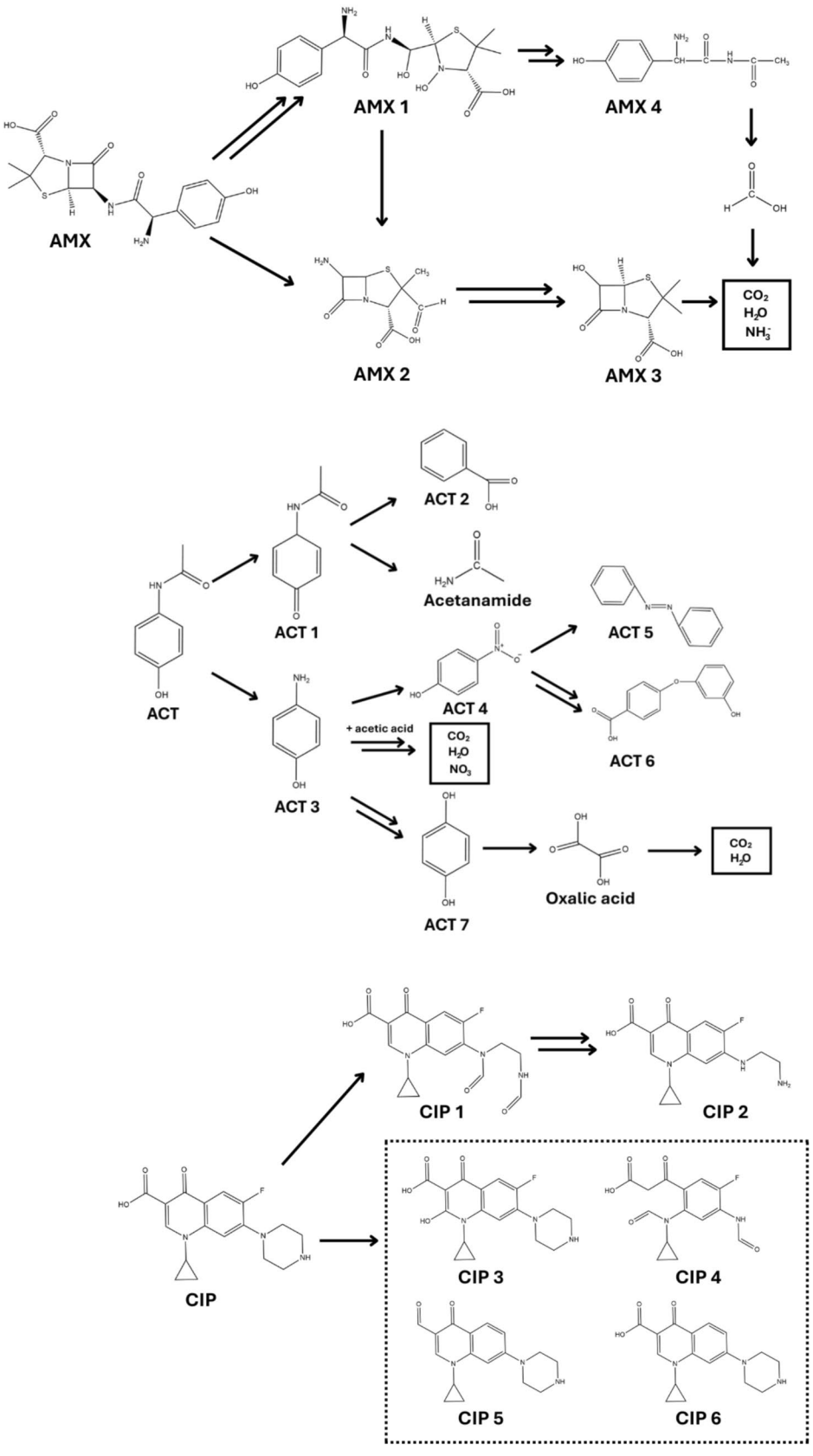

Fig. 4 Proposed degradation pathways of AMX, ACET and CIP via ozonation (Anjali and Shanthakumar 2022)

despite its effectiveness, such as the need for large volumes of Fe^{2+} ions (often from the $FeSO_4$ salt), and the requirement for additional Fe^{2+} ion and sludge treatment. The heterogeneous photo-Fenton reaction involves solid catalysts such as iron oxides, iron-based composites and iron-based semiconductors (Gou et al. 2021). The iron-based heterogeneous photo-Fenton process has demonstrated superiority over its homogeneous counterpart, due to the convenience of catalyst recycling and the ability to operate effectively across a broad pH spectrum. Furthermore, both Fenton and photo-Fenton operate with simpler technology and fewer specialized equipment requirements.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH + HO \quad (2)$$

$$2Fe^{3+} + Fe^{0} \rightarrow 3Fe^{2+} \quad (3)$$

$$Fe(OH)^{2+} + h\nu \rightarrow Fe^{2+} + OH \ \lambda < 580nm \quad (4)$$

The current trend shows that Fenton oxidation has been induced using zero-valent iron (ZVI), which is a heterogeneous reaction. The advantages of ZVI-Fenton reaction compared to conventional Fenton are (i) both reduction and oxidation processes occur on the surface of ZVI by the electron transfer, (ii) recycling of ZVI particles as ferric (Fe^{3+}) iron into Fe^{2+} species (Eq. 2), (iii) production of less residue and (iv) cost-effectiveness (Li et al. 2022). The ZVI-Fenton reaction in the presence of UV light and visible light has been also gaining great attention.

The degradation of ibuprofen in real wastewater collected from WWTP was studied by ZVI nanoparticle–Fenton treatment. It was found that at pH 3 and pH 4, the highest removal resulted in the presence of 0.01 g/L ZVI and 50 µM H_2O_2 (Minella et al. 2019). The degradation of ibuprofen was reported to be dependent on the pH and loadings of ZVI and negligible degradation was found at the natural pH of the wastewater (pH 7.6). Another study also reported that the pH and loadings of ZVI greatly affect the removal of antibiotics cefazolin, imipenem and vancomycin from two types of urban wastewater samples (Furia et al. 2021). Although ZVI alone did not produce any removal, ZVI-Fenton demonstrated complete removal within 90 min in the presence of 0.02 g/L ZVI and 400 µmol/L H_2O_2 at pH 5 for both wastewaters. This ZVI-Fenton technique also did not work at the natural pH of the wastewater (pH 7.5–7.8) as reported by Minella et al. (2019). pH 5 can be considered effective in terms of cost and the amount of acid (H_2SO_4) needed for pH adjustment in contrast to the classical Fenton, which requires pH 3 for effective degradation. Li et al. (2022) investigated the removal of triclosan and paracetamol (and two other personal care products) using iron powder and UVC-Fenton (pZVI). The authors claimed that iron powder is cheaper and can be easily produced compared to nanoparticle-ZVI. The application of UVC-Fenton (pZVI) effectively attained complete removal after 30 min of treatment in the presence of a molar ratio of H_2O_2/pZVI of 2.0 and pZVI concentration of 22.4 mg/L at a pH of 3.0.

Typically, Fenton and photo-Fenton are known to be effective under acidic conditions (pH 2–3), which appears to be the major downside of these processes, although related studies have been reported to be efficient in removing the pharmaceutical compounds (Całus-Makowska et al. 2025). For example, solar photo-Fenton was reported to be effective in removing mixed pharmaceutical pollutants with > 99% removal under optimized conditions (pH 3, Fe^{2+} 0.04 mM, H_2O_2 4 mM) (Anjali and Shanthakumar 2024). An acute and chronic toxicity study performed on the transformation products was observed to fall under "harmless category" (EC50 > 100 mg/L according to European Union Commission) (EU directive 93/67/EEC). Carbamazepine degradation was conducted via photo-Fenton using solar simulator by Parra-Enciso et al. (2023). Under optimal conditions (pH 3) determined using central composite design (CCD), 96.4% removal of CBZ was achieved with 0.750 mg/L H_2O_2 and 0.2 mg/L Fe^{2+}. Degradation product analysis performed successfully reported a new product, 2,2′-aminodibenzaldehyde (CBZ 1 in Fig. 5). Figure 5 elucidates the proposed degradation pathway for carbamazepine.

Despite the positive outcomes, there has been continuous effort to perform these processes at near-neutral pH to make this approach more attractive. Photo-Fenton and electro-Fenton processes were tested at the laboratory scale for pharmaceutical pollutant removal from real urban wastewater, under

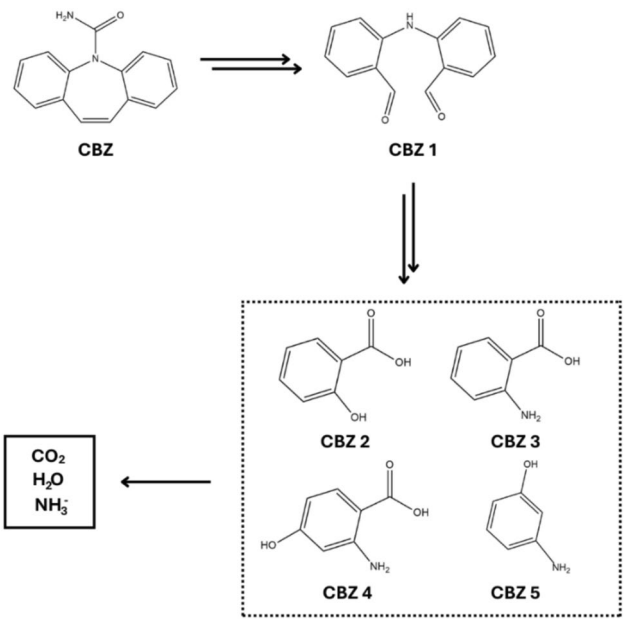

Fig. 5 Proposed degradation pathway of carbamazepine via photo-Fenton

its natural circumneutral pH conditions (Jiménez-Bambague et al. 2023). The study revealed that the electro-Fenton process achieved the highest removal efficiency of 81.1% for these compounds. Nevertheless, some compounds, like carbamazepine, increased in concentration after the photo-Fenton process, indicating potential inefficiencies. A study by Perini et al. (2018) attempted to evaluate the treatment of UVC-assisted photo-Fenton at neutral pH (pH 7.4) for hospital effluent spiked with amoxicillin, ciprofloxacin, sulfathiazole and sulfamethazine in the presence of citric acid serving as a ligand. Among the investigated systems, the UVC/H_2O_2/FeCit system resulted in complete degradation of all antibiotics after 90 min of treatment time with 82% consumption of the H_2O_2 (initial concentration used = 500 µM). In view of the finding, the environmentally friendly chelating agent, ethylenediamine-N,N'-disuccinic acid (EDDS), was studied in solar photo-Fenton study at pH 7 for the removal of pharmaceuticals in raw hospital wastewater sampled from a local hospital in Brazil (Cuervo Lumbaque et al. 2021). EDDS possesses the desirable characteristics of being highly biodegradable, non-toxic under environmental conditions and can maintain iron species in solution across a wide pH range. Molar ratio of Fe^{3+}-EDDS (1:2) $(Fe^{3+}]_0$ = 15.35 mg/L and $[H_2O_2]_{total}$ = 230 mg/L resulted in > 70% removal of pharmaceutical compounds.

The photo-Fenton process mediated by ferrioxalate has been also explored to operate under pH 7 and under visible radiation. Marchetti and Azevedo (2020) applied response surface methodology as a tool to optimize the removal of NSAIDS namely salicylic acid, ketoprofen, diclofenac and paracetamol by photo-Fenton mediated with ferrioxalate with UV-LEDs. Although complete removal below detection limits was attained after 25 min using LED power of 1.768 W, Fe^{3+} of 15.0 mg/L and H_2O_2 47.6 mg/L, the COD and DOC removals were only 40% and 30%, respectively. A phytotoxicity test against lettuce seeds, *Lactuca sativa*, did not reveal any sign of ecotoxicity. In addition, solar photo-Fenton is also known to be environmentally friendly and cost-effective and uses natural solar light to generate the ·OH radicals needed to drive the treatment. A study by Foteinis et al. (2018) reported that treating real pharmaceutical effluent using solar photo-Fenton mediated by ferrioxalate using a semi-industrial autonomous solar compound parabolic plan can be sustainable due to a low contribution to the carbon footprint estimated using the IPCC 2013 impact assessment method. Another photo-Fenton experiment carried out using a compound parabolic collector (CPC) under direct solar radiation for the degradation of amoxicillin and paracetamol revealed that the percentage degradation and mineralization measured as TOC is controlled by the amount of H_2O_2 added in the reaction system and type of water, whether simulated water or WWTP effluent (Hinojosa Guerra et al. 2019). Besides, direct solar radiation, a simulated solar-mediated photo-Fenton process, is also common at a laboratory scale. For example, this process was reported to effectively remove non-steroidal anti-inflammatory pharmaceuticals namely ketoprofen, meloxicam and tenoxicam (each 10 mg/L) at pH 3–4 in the presence of 400 mg/L of H_2O_2 and 1.75 mg/L Fe^{2+} with COD removal of > 98% (de Melo Santos et al. 2020). Despite this process, optimizing a suitable photoreactor and reagents for solar photo-Fenton to reduce the consumption of H_2O_2 remains a major challenge that needs attention.

The development of heterogeneous Fenton catalysts using Fe-based materials is also becoming a popular strategy for pharmaceuticals removal, particularly catalysts that can harness visible light within the solar energy spectrum (Ortega-Moreno et al. 2022; Qiu et al. 2022). Studies have demonstrated the efficiency of such catalysts for the degradation of pharmaceuticals in wastewater such as the removal of oxytetracycline via erbium ferrite/graphitic carbon nitride ($ErFeO_3$/g-C_3N_4), which resulted in 99.7% removal using an LED light source (Wang et al. 2022). Typically, the remarkable performance of heterogeneous (photo) Fenton catalysts has been associated with various factors such as morphology, specific surface area, rapid electron transfer and Fe (II) regeneration. A review by Usman et al. (2023) has summarized the latest findings on the heterogeneous catalysts for pharmaceutical removal via adsorption and Fenton-based processes. The combination of photo-Fenton with other AOPs such as photocatalysis has shown promising findings in the removal of pharmaceutical compounds (Mishra et al. 2024). For instance, the combined photo-Fenton and photocatalysis process significantly enhanced the degradation rate of pollutants in wastewater from a pharmaceutical industry in India, achieving a first-order rate constant of 0.422 min^{-1}, compared to 0.195 min^{-1} for photocatalysis and 0.125 min^{-1} for photo-Fenton alone (Talwar et al. 2021).

Based on these studies, Fenton and photo-Fenton-based processes are highly effective in degrading pharmaceutical pollutants in wastewater, offering advantages such as high degradation efficiency, cost-effectiveness and environmental sustainability, particularly when integrated with solar or UV light sources. The development of heterogeneous catalysts has further enhanced their applicability by improving catalyst recyclability, expanding the operational pH range and reducing sludge production. However, challenges remain, including the need for acidic conditions, high H_2O_2 consumption and process optimization to achieve efficient degradation at near-neutral pH.

Direct UV and other UV-based processes

The UV-direct process employs UV irradiation to excite chemical compounds (Legrini et al. 1993). While UV has traditionally been utilized for disinfecting drinking water,

there have been recent studies focused on removing pharmaceutical compounds from aqueous solutions (Chowdhury et al. 2020; Ping et al. 2021). The effectiveness of this process is largely determined by two key factors: the molar absorption coefficient of the molecule being treated and the quantum yield (Chowdhury et al. 2020). Although the UV-direct process can degrade pharmaceutical compounds, it has limited capacity for mineralization, resulting in the presence of intermediate or degradation compounds in the treated solution (Wang et al. 2019). The arising toxicity and environmental risks are associated with the UV photolysis of the transformation products from the parent compound degradation as observed by Ke et al. (2023) during the UV photolysis of glucocorticoids. Likewise, a study by Ping et al. (2021) reported that acute toxicity levels of atenolol (ATO) bezafibrate (BZB) and metoprolol (MET) were found to increase after photolysis because of degradation products formed suggesting that assessment of ecological risk of compounds formed on degradation via photolysis is crucial. Degradation of MET, atenolol (ATE), BZB and ATO was inhibited by the presence of NO_3^-, Cl^-, HCO_3^- and humic acid because of competition for photons or light-shielding effect. On the other hand, the UV/H_2O_2 method employs H_2O_2 irradiation to produce ·OH radicals, which are potent oxidants that improve the effectiveness of oxidation and increases the mineralization rate (Pai and Wang 2022; Somathilake et al. 2019). A study revealed that positioning UV/H_2O_2 after activated sludge treatment as a polishing step resulted in a better removal efficiency for various pharmaceutical compounds from wastewater, including analgesics and anti-inflammatories, antibiotics, psychiatric and other pharmaceuticals (Mir-Tutusaus et al. 2021). This finding has challenged the perspective of those who reported that biological treatment usually appears prior to UV/H_2O_2 in coupled treatments.

The degradation of ciprofloxacin compared by UV photolysis and UV/H_2O_2 found that the latter process was able to completely remove 10 mg/L of the compound in the presence of 100 mmol/L of H_2O_2 within 40 min of reaction time at an initial pH 7 (Mondal et al. 2018). Three anti-COVID-19 drugs, RDV, DEX and HCQ in ultrapure water were investigated via UV and UV/H_2O_2 by Huang et al. (2022). The degradation rate of all three drugs was higher for UV/H_2O_2 than for UV alone. DEX demonstrated the highest degradation efficiency and higher mineralization rate, which led to less TPs and lower toxicity, based on the bacterial luminescence rate. The degradation of RDV via UV/H_2O_2 resulted in eight degradation products, which were detected using high-performance liquid chromatography quadrupole time-of-flight mass spectrometry (HPLC-QTOF-MS) (Fig. 6).

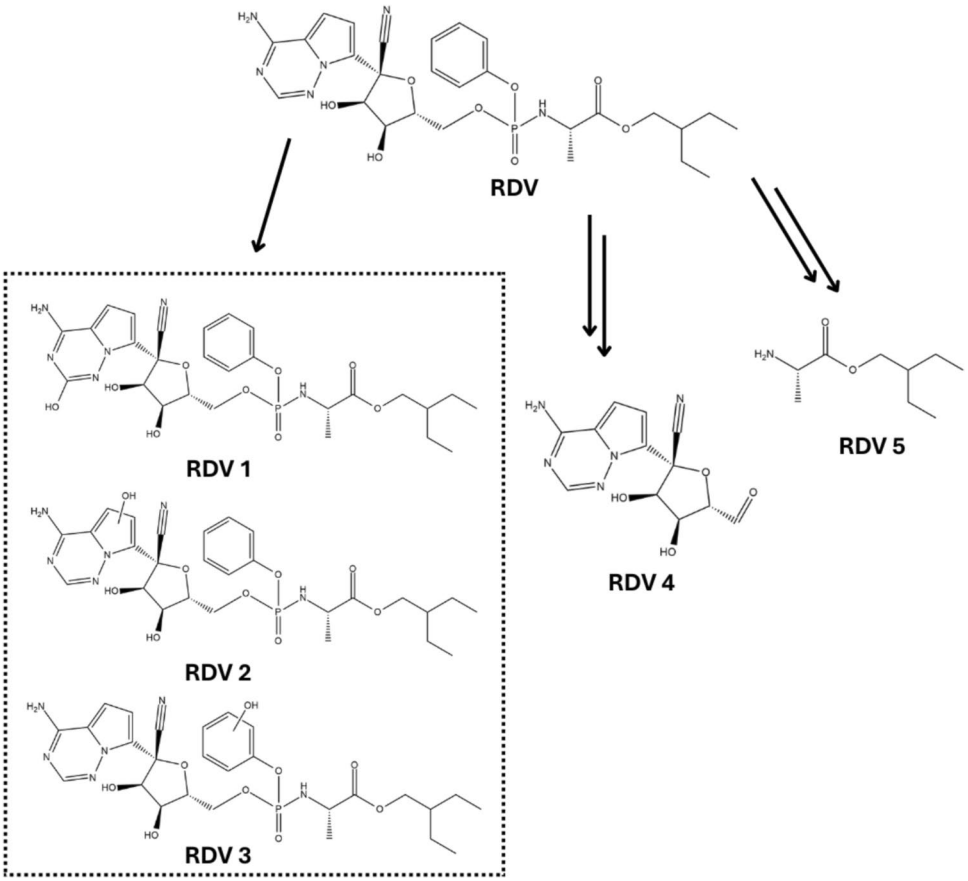

Fig. 6 Proposed degradation pathway of RDV via UV/H_2O_2

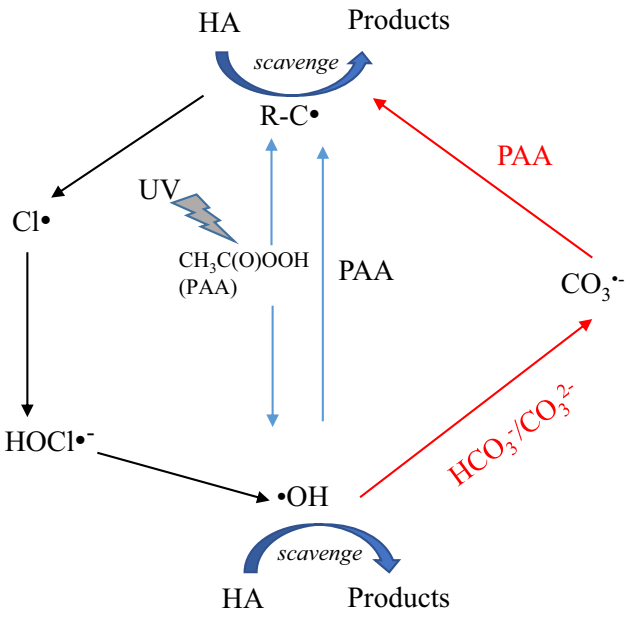

Fig. 7 Naproxen degradation via UV/PAA process in the presence of HA, Cl^- and CO_3^{2-}/HCO_3^- (redrawn from Chen et al. 2019) (Reprinted with permission)

Due to a variety of reasons, peracetic acid (PAA, CH_3CO_3H) is becoming increasingly popular as a complement to UV, in addition to H_2O_2 (Chen et al. 2019; Ghanbari et al. 2021). UV/PAA has several advantages over UV/H_2O_2, including the generation of multiple radicals, such as acetyloxyl radicals, methyl radicals, peroxyl radicals and acetylperoxyl radicals (Rizzo et al. 2019). While these radicals are generally less reactive than HOC, they can still offer new reaction mechanisms and pathways. A recent study investigated the combined effect of UV and PAA as a novel approach for treating pharmaceutical contaminants in municipal wastewater (Hollman et al. 2020). All four pharmaceuticals, sulfamethoxazole, fluoxetine, carbamazepine and venlafaxine, were completely degraded by UV/PAA following pseudo-first-order kinetics within 40 s, 300 s, 500 s and 400 s, respectively. To investigate the effect of a water (ultrapure) matrix containing of CO_3^{2-}/HCO_{3-}, humic acid (HA) and Cl^-, a study attempted to degrade naproxen by UV/PAA process (Chen et al. 2019). A minimal inhibitory effect was observed for all water matrices in the UV/PAA process, due to the formation of different radical species such as R–C·, and ·OH (Fig. 7). The reaction mechanism pathway of UV/PAA is not well-established compared to the straightforward reaction of UV/H_2O_2. Table 3 compares the characteristics between UV/PAA and UV/H_2O_2 under UV photolysis.

UV/chlorine and UV/sulfate radical-based AOPs have been also explored as tools for pharmaceutical removal in water treatment. The chlorine radical (Cl·) has a standard potential of 2.4 V (Khajouei et al. 2022), while sulfate radical ($SO_4^{\cdot-}$) has a higher oxidation capability than ·OH radical (E_0 = 2.65–3.1 V) (Liu et al. 2020). A study which compared the efficiency between UV/H_2O_2 and UV/PS (persulfate) for the degradation of ofloxacin and levofloxacin found that the latter was more efficient in removal and mineralization of these antibiotics with second-order rate constants of 2.19×10^{10} and 2.66×10^{10} $M^{-1}s^{-1}$, respectively (Liu et al. 2020). Cobo-Golpe et al. (2022) compared the efficiency of conventional UV and chlorination processes with UV/H_2O_2 and UV/chlorine for the degradation of tramadol. Among the studied processes, UV/chlorine treatment in the presence of 10 mg/L of chlorine was superior in removing > 70% of tramadol in wastewater samples. Despite that, TOC removal was almost negligible for both UV/H_2O_2 and UV/chlorine in ultrapure water samples, spiked with 0.5 mg/L of tramadol and 10 mg L^{-1} and 5 mg L^{-1} for free chlorine and H_2O_2, respectively. It was found that the presence of inorganic species such as chlorides, nitrates and ammonium in water affected both UV/H_2O_2 and UV/chlorine treatments at different rates. The performance of UV/H_2O_2 and UV/chlorine on the removal of pharmaceutical compounds has been reported to vary with the type of compound, number of radicals generated and type of water effluent or water quality parameters (Ngumba et al. 2020).

Based on these studies, UV-based processes are promising technologies for the degradation of pharmaceutical

Table 3 Comparison between the characteristics of H_2O_2 and PAA under UV photolysis (Chen et al. 2019; Ghanbari et al. 2021; Ngumba et al. 2020; Pandis et al. 2022; Rizzo et al. 2019; Wang et al. 2019)

	UV/H_2O_2	UV/PAA
Photolysis/cleavage	$H_2O_2 + UV \rightarrow 2{\cdot}OH$ (Cleavage of H_2O_2)	$CH_3CO_3H + UV \rightarrow CH_3COO{\cdot} + {\cdot}OH$ (photolysis of PAA) $CH_3COO{\cdot} \rightarrow CH_3{\cdot} + CO2$ $\cdot OH + \cdot OH \rightarrow H_2O_2$
Characteristics	Unstable and tends to undergo inefficient decomposition	Clear, colourless, highly corrosive, strong pungent odour
Merits	No sludge production, commercially viable, simultaneous use for disinfection and removal	Produces several radicals (hydroxyl and acetoxyl radicals), high efficiency for degradation and disinfection
Demerits	High energy and amount of reagent (H_2O_2) needed to generate sufficient ·OH radical, high cost	Produces organic residuals (acetic acid) in solution

contaminants in water treatment. These processes offer several advantages, including high degradation efficiency, fast reaction rates and the potential for complete removal of target pollutants. Additionally, some UV-based processes, such as UV/PAA, exhibit resilience against matrix effects, making them viable for real wastewater applications. However, these processes also present challenges, such as incomplete mineralization, the potential formation of toxic byproducts and sensitivity to water matrix components (e.g. nitrates, chlorides, humic acids), which can inhibit degradation efficiency. The high energy consumption of UV irradiation and the need for optimal reagent dosing further complicate large-scale applications.

Sonolysis

Sonolysis is an AOP that utilizes HO· radicals to break down various organic pollutants. This process occurs when sound waves at specific frequencies create cavitation bubbles in the liquid. These bubbles grow and then implode violently, producing extreme conditions of high temperature (around 4200 K) and pressure (up to 975 bar), creating "hot spots". These conditions enable the decomposition of water molecules, leading to the formation of HO· radicals that effectively degrade organic contaminants (Camargo-Perea et al. 2020). Compared to other AOPs, ultrasound has distinct advantages, including selective degradation and physical effects, being environmentally clean as it does not require addition of chemicals in the treatment system and it does not generate waste like in Fenton and photo-Fenton processes (Serna-Galvis et al. 2019; Camargo-Perea et al. 2020). This technique, however, is constrained by substantial energy consumption and long reaction times.

Studies have investigated the performance of sonolysis in the degradation of pharmaceuticals, particularly single pharmaceuticals (Khalid et al. 2022). The effect of ultrasound on the simultaneous removal of pharmaceuticals in mixed systems is rather scarce. Stucchi et al. (2020) made a comparison between the degradation of amoxicillin and acetaminophen in water as individual and mixed components. Only 15% of acetaminophen was degraded, while no degradation took place for amoxicillin, when they were degraded simultaneously by ultrasound (40 W; 20 kHz). In contrast, 22% of amoxicillin was removed in the individual system, while acetaminophen was observed to be more resistant with only 10% removal at the same ultrasound power of 40 W.

Studies have also investigated the application of both low frequency (Karim and Shriwastav 2021; Parra-Enciso et al. 2022) and high frequency (Abdelhay et al. 2020; Serna-Galvis et al. 2019) to the degradation of pharmaceutical compounds. High frequency of 578 kHz and 23.8 W was able to degrade 32% of the antibiotic meropenem, within 60 min of treatment time. Another study which investigated the effect of high frequency ultrasound (375 kHz; 88 W/L) on the effluents from municipal WWTP in Columbia found that the elimination levels were dependent on the concentration of pharmaceuticals and their hydrophobicity (Serna-Galvis et al. 2019). Pollutants that are more hydrophobic in the effluent degrade faster as they are in closer proximity to cavitation bubbles. This makes them more vulnerable to reactions with HO· radicals. Furthermore, the presence of suspended solids in the effluent also somewhat affected the removal of a few pharmaceutical compounds such as ciprofloxacin, norfloxacin, diclofenac and sulfamethoxazole. A recent study investigated the effect of multiple frequencies (dual frequency) to enhance the removal of paracetamol and at the same time to reduce the energy consumption and lengthy reaction time for this removal (Zare et al. 2023). It was found that the investigation of synergistic effect of dual frequencies enhanced the degradation of paracetamol.

Acoustic intensity or power and acoustic frequency play important roles in the sonochemical degradation of pharmaceuticals (Camargo-Perea et al. 2021; Gasmi et al. 2023). A study investigated the impact of three ultrasonic frequencies (585, 860 and 1140 kHz) with a fixed acoustic intensity of 4.3 W/cm^2 on the degradation of 10 mg/L furosemide (Gasmi et al. 2023). The degradation trend decreased with the increasing frequency with 585 kHz being optimum. Despite the complete removal of furosemide within 60 min of treatment, the TOC level was only 25% after 4 h of treatment. The effect of power densities also governs the degradation rate of pharmaceutical compounds. For example, degradation of trimethoprim in ultrapure water was observed to increase from 0.0177 to 0.0355 min^{-1} with the increasing power density from 20 to 60 W/L at a low frequency of 20 kHz due to the increased level of acoustic cavitation, which generated more HO· radicals in the system (Arvaniti et al. 2020).

Based on these studies, sonolysis is a promising AOP for the degradation of pharmaceutical contaminants in water treatment. It offers several advantages, including selective degradation, effective breakdown of hydrophobic pollutants and improved efficiency, when dual-frequency ultrasound is applied. Unlike other AOPs, such as Fenton and photo-Fenton processes or catalytic oxidation technologies, ultrasound does not produce chemical residues or sludge, making it an environmentally cleaner option. Additionally, integrating ultrasound with other AOPs or conventional water treatment methods can significantly enhance removal and mineralization rates, leading to more efficient pollutant degradation. However, while such hybrid approaches improve treatment outcomes, they often come with higher economic costs, compared to using ultrasound alone.

Electrochemical processes

The high potential for degrading refractory pollutants, environmental friendliness, absence of sludge production, in situ generation of oxidants and the use of electrons as a green reagent have made the electrochemical process a subject of great interest for the removal of pharmaceuticals from wastewater. Despite these advantages, the electrochemical process faces challenges such as the high cost of specialized electrodes, such as boron-doped diamond (BDD) anodes, which limits their widespread use, dependence on conductive electrolytes and high energy consumption.

There are two primary categories of electrochemical-based AOPs: one relies on the direct transfer of electrons between pollutants and electrodes (direct anodic oxidation), while the other is based on the creation of oxidants through electrochemical means (indirect oxidation) (Souza et al. 2022). In the direct method, organic compounds undergo transfer reactions directly on the anode surface with the aid of electrons to facilitate their oxidation. On the other hand, the indirect method relies on highly oxidizing agents, such as reactive oxygen species (·OH) and chlorine active species (ClO^- at pH > 8, HClO in the pH range 3–8 and Cl_2 at pH < 3), which are produced electrochemically at the anode surface to facilitate the oxidation process (López Zavala et al. 2020).

Boron-doped diamond (BDD) anodes have gained significant popularity in water treatment applications owing to their favourable chemical and electrochemical stability, inert surface, low adsorption capabilities, prolonged lifespan and broad potential range (Donoso et al. 2021). Several studies have applied this type of anode for pharmaceutical abatement. For example, current density has been reported as the crucial parameter in determining the efficiency of Nb/BDD anode (and Ti cathode) for the removal of 11 pharmaceuticals in hospital effluent (Ouarda et al. 2019). It was found that a high current density of 35.4 mA/cm^2 resulted in the highest abatement percentages ranging from 50 to 80% within 120 min of treatment. Another study also applied the Nb/BDD anode for the simultaneous degradation of sulfamethoxazole, propranolol and carbamazepine, but compared oxygenated and non-oxygenated conditions (García-Espinoza and Nacheva 2019). In the spiked secondary effluent, a lower degradation kinetic rate (first-order) was obtained in the presence of oxygen compared to its absence due to the competitive reactions occurring between ROS and other oxidants, such as active chlorine. Sulfamethoxazole and propranolol were degraded completely within 30 min in the presence of oxygen, compared to carbamazepine which took a longer time at a 2.5 A current and without the addition of electrolyte. In contrast, TOC and COD removal was enhanced by oxygen with 67% and 89% removal, respectively. The degradation pathway of the simultaneous degradation of all three pharmaceutical compounds, sulfamethoxazole, propranolol and carbamazepine was reported to be driven by bond breaking, hydroxylation, deamination and also desulfunation (Fig. 8).

Despite the positive performance of BDD anodes, studies have also indicated that such anodes can be costly, which makes them uneconomical (Giannakopoulos et al. 2022). Likewise, electrodes such as doped SnO_2 and PbO_2 lead to Pb leaching or contamination and instability. As such, alternative or cheaper electrodes are also a research focus. Teng et al. (2020) investigated a Ti suboxide mesh anode as an alternative owing to its high conductivity, good stability and low cost for the degradation of sulfadiazine. A complete removal of sulfadiazine was achieved within 60 min of electrolysis using a current density of 10 mA/cm^2 and Na_2SO_4 electrolyte compared to $NaNO_3$ (92%) and $NaClO_4$ (88%) electrolytes. TOC removal of 79% was obtained under the same condition compared to commercial anodes, Pt and PbO_2, which produced lower removals of 63% and 43%, respectively, because of higher oxygen evolution potential. A Pt–SnO_2/Ti anode was used for the degradation of diclofenac (Giannakopoulos et al. 2022).

Overall, electrochemical processes are a promising option for pharmaceutical removal from wastewater, offering environmental benefits and high efficiency, but the economic factors related to electrode materials and energy consumption need to be addressed for broader adoption.

TiO_2 photocatalysis

TiO_2-mediated heterogeneous photocatalysis appears to be one of the most promising destructive methods for the degradation of pharmaceutical compounds. This is due to the multiple advantages of TiO_2, including its non-toxicity, low cost, high photoreactivity and photostability (Ruziwa et al. 2023). Typically, photocatalysis begins when TiO_2 interacts with light of sufficient energy (< 387 nm) and is subsequently followed by the excitation of electrons from the filled valence band (VB) to the vacant conduction band (CB), leaving positive holes in the valence band. The photo-induced holes (h_{VB}^+) react with organic compounds to form intermediate products, which will be then decomposed into CO_2 and H_2O. To ensure a successful production of reactive oxidants, two events must occur simultaneously during the photocatalytic reaction. The first event involves the photo-induced holes diffusing to the TiO_2 semiconductor surface and followed by the second event, which is the reaction of photo-induced holes with water molecules to generate ·OH radicals (Lee and Park 2013). The ·OH radicals are responsible to oxidize and mineralize pharmaceutical organic pollutants into intermediate products, mineral salts and final degradation products, CO_2 and H_2O.

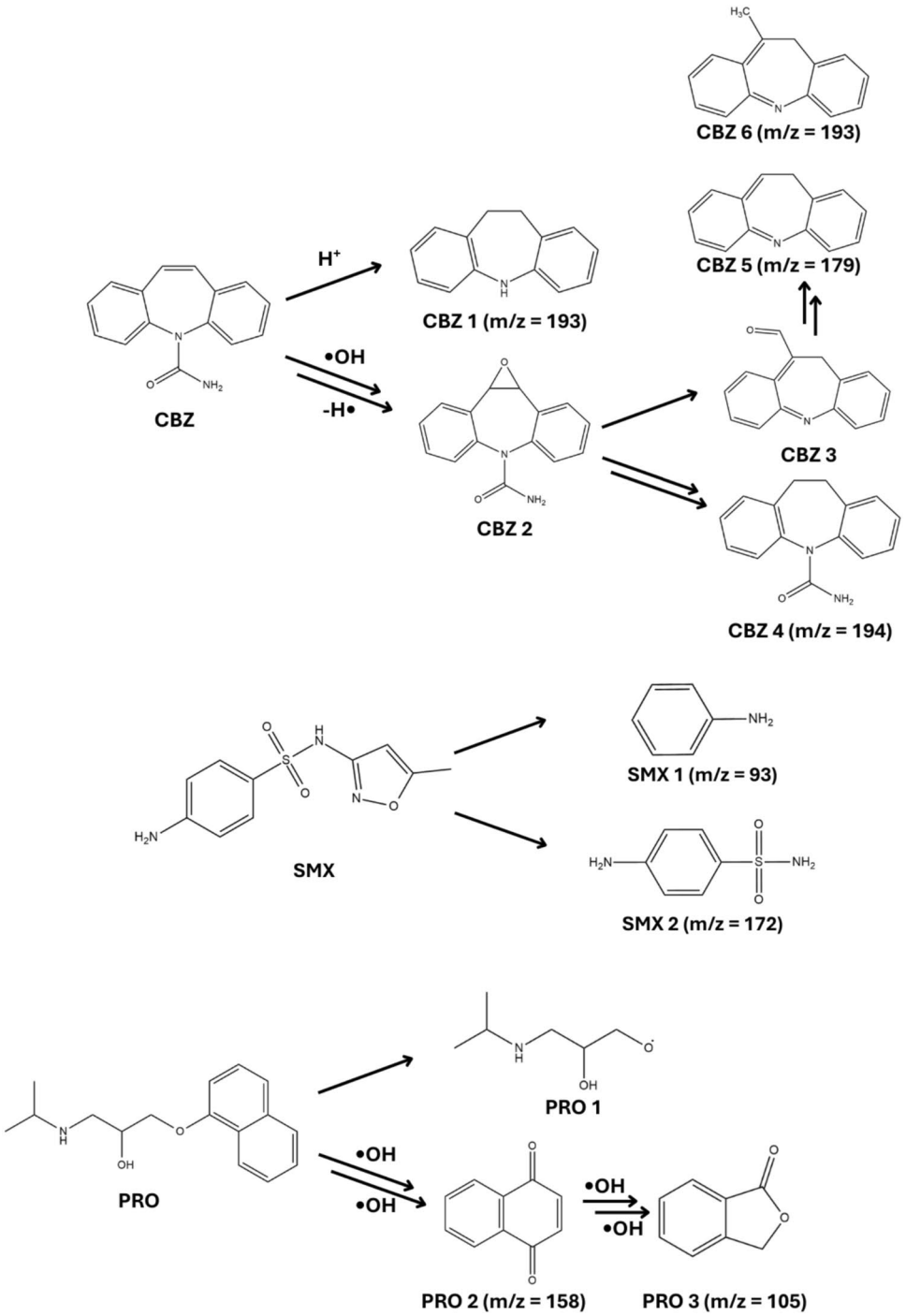

Fig. 8 Simultaneous degradation pathways of CBZ, SMX and PRO sulfamethoxazole, propranolol and carbamazepine via electrochemical oxidation

Photocatalytic degradation of pharmaceuticals via TiO_2 semiconductors is one of the most superior methods. The application which begins with various types of pure or pristine TiO_2 has led to the evolution of engineered TiO_2-based nanomaterials, using numerous approaches and materials. In this section, the performance of both commercially available TiO_2 and synthesized TiO_2-based materials related to the degradation of pharmaceuticals will be discussed.

The effects of parameters were investigated to relate the performance of TiO_2 or TiO_2-based nanomaterials or photocatalysts to the degradation of pharmaceuticals. Modification of pristine TiO_2 becomes necessary due to being severely constrained by the rapid recombination of photogenerated electron holes or low photonic efficiency, inadequate ability to capture sunlight and the challenge of isolating the photocatalysts from aqueous solutions. The commonly examined parameters are the concentration of

TiO_2 or the TiO_2-based photocatalysts, initial concentration of pollutant, types of water matrix, pH of solution or water and presence of water matrix constituents. Current progress shows that various studies have been performed using commercially available or synthesized TiO_2 and synthesized TiO_2-based nanomaterials or advanced materials for the degradation of various pharmaceutical compounds. Figure 9 illustrates applications of pure TiO_2 and TiO_2-based materials for the degradation of pharmaceuticals.

Commercial TiO_2

Commercial TiO_2 such as Aeroxide P25 TiO_2, Hombikat UV l00 and Merck TiO_2 have been applied to the degradation of pharmaceuticals, under both artificial light sources and natural sunlight owing to their photocatalytic activity and physico-chemical properties (e.g. optical properties, surface properties). The performance of commercial TiO_2 on the degradation of pharmaceuticals has been reported in various studies (Table 4). For example, Cabrera-Reina et al. (2019) have investigated the degradation of imipenem and meropenem antibiotics using Aeroxide P25 TiO_2, under natural solar irradiation using a CPC photoreactor. The photocatalytic performance on the initial reaction rates was influenced by the type of water matrix used, whereby river water and simulated WW effluent resulted in decreased reaction rates compared to ultrapure water for both antibiotics using 50 mg/L TiO_2. Ponkshe and Thakur (2019) screened the performance of four TiO_2 candidates namely Aeroxide P25, Hombikat UV 100, Kronoclean 7000 and Merck TiO_2 related to the degradation of atenolol and propranolol and found that Aeroxide P25 appeared to be superior, producing the highest first-order degradation rate (0.056 min^{-1} for propranolol and 0.026 min^{-1} for atenolol) compared to its counterparts. Removal of antibiotics from real livestock wastewater using Aeroxide P25 TiO_2 revealed that the degradation efficiency is governed by the structure of the antibiotic compound as variations in removal percentages were observed, from complete removal to < 70% (Park et al. 2022). A recent study Lykos et al. (2023) explored the degradation kinetics of metronidazole using commercial TiO_2 P25 at a laboratory scale (using a solar simulator) and pilot scale (CPC reactor) (Fig. 10a). Identification of toxicity products revealed a total of seven products using the solar simulator with three of them also being detected at pilot scale (TP4, TP5 and TP7) (Fig. 10b). Toxicity study using *Vibrio fischeri* revealed that fewer toxic products were formed after exposure to solar irradiation in the presence of TiO_2 P25.

Based on the studies which have applied commercial TiO_2 for the degradation of pharmaceuticals, it was observed that the treatment kinetics vary with the type of commercial TiO_2 used in the study and their effects on the studied APIs. Most studies conducting the degradation experiments in suspension using and characterizations data of the material are scarce, limiting a thorough comparison based on the TiO_2 properties. Only a few studies performed characterization analyses for the chosen commercial TiO_2, but most of them were not comprehensive. Akter et al. (2022) determined the X-ray diffraction (XRD) pattern of TiO_2 which confirmed the presence of the anatase phase (Fig. 10c) while Park et al. (2022) performed XRD, TEM, SEM and FTIR analyses on the Aeroxide P25 in which XRD revealed the presence of a combination of rutile and anatase phases. However, although related studies emphasized the degradation of parent compounds, little attention has been given to the identification of arising degradation products, mineralization and monitoring of the toxicity level in the degraded solution.

Modified TiO_2-based materials

Albeit TiO_2 nanoparticles (TiO_2 NPs) are a promising material for photocatalytic degradation of pharmaceuticals, this

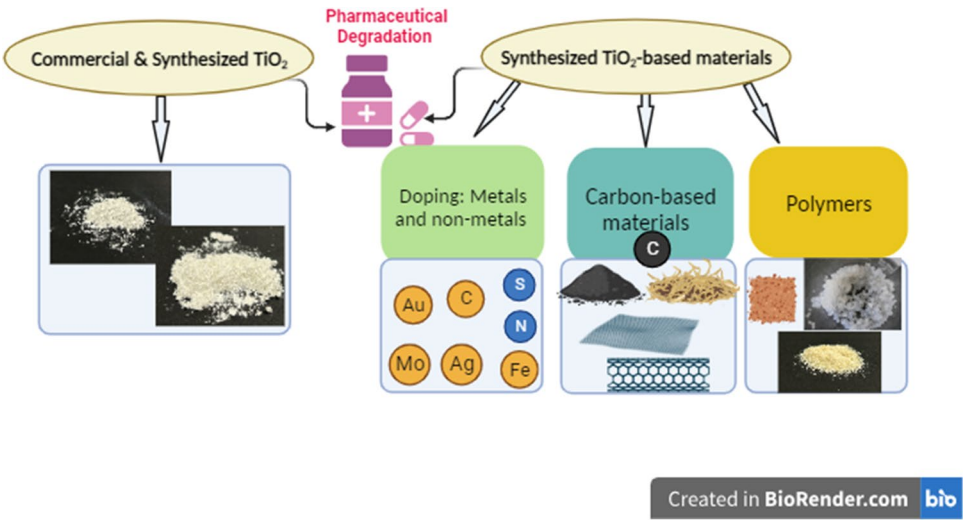

Fig. 9 Applications of pure TiO_2 and TiO_2-based materials for the degradation of pharmaceuticals (created using BioRender)

Table 4 Comparison of commercial TiO_2 used in pharmaceutical degradation studies

Commercial TiO_2 and source	Pharmaceutical compound and percentage removal (%)	Optimized conditions	Properties			Reference
			Crystal form	BET (m^2/g)	Particle size (nm)	
TiO_2 P25 Evonik (Essen, Germany)	Metronidazole (MTZ) (91% under CPC reactor and 100% under lab condition)	10 mg/L of MTZ and 100 mg/L of TiO_2 P25 (lab scale) 10 µg/L of MTZ and 300 mg/L of TiO_2 P25 (pilot scale)	80% anatase/20% rutile	56	20–30	Lykos et al. (2023)
Aeroxide P25 TiO_2 Evonik Industries	Imipenem (75% after 60 min) and meropenem (75% after 45 min) under solar radiation	10 mg/L TiO_2 and 500 µg/L of imipenem and 50 µg/L meropenem	NA	35–65	NA	Cabrera-Reina et al. (2019)
Aeroxide P25 NA	Ceftiofur, clopidol, enrofloxacin, erythromycin, florfenicol, lincomycin, oxytetracycline, sulfamethoxazole etc. (> 90% removal after 60 min)	TiO_2: 1.0 g/L Total compound concentration: 350 µg/L (17 antibiotics compounds)	Anatase/rutile	35–65	20	Park et al. (2022)
Aeroxide P25 TiO_2 Evonik Industries	Propranolol (PR) (96%) atenolol (AT) (94%)	TiO_2: 0.03 g/L PR: 5×10^{-5} M AT: 2×10^{-4} M	75% anatase/25% rutile	50	21	Ponkshe and Thakur (2019)
TiO_2 Inframet Advanced Materials (USA)	Sulfamethoxazole (SMX) (97% in 360 min), metronidazole (MNZ) (100% in 600 min) and ciprofloxacin (CIP) (100% in 600 min)	SMX: 0.7 g/L TiO_2 and 5 mg/L SMX MNZ: 0.7 g/L TiO_2 and 80 mg/L MNZ CIP: 0.7 g/L TiO_2 and 80 mg/L CIP	99.9% anatase	NA	25	Akter et al. (2022)
P25 TiO_2 Degussa AG Quimidroga SA	Tetracycline (TET) (99.75% after 4.2 h), ciprofloxacin (CIP) (99.90% after 3.5 h), sulfadiazine (SDZ) (99.61% after 7.1 h) and sulfamethoxazole (SMX) (98.84% after 16.5 h)	TiO_2: 1 g/L Initial concentration: 20–1000 µg/L	NA	50 ± 15	21	Zambrano et al. (2022)

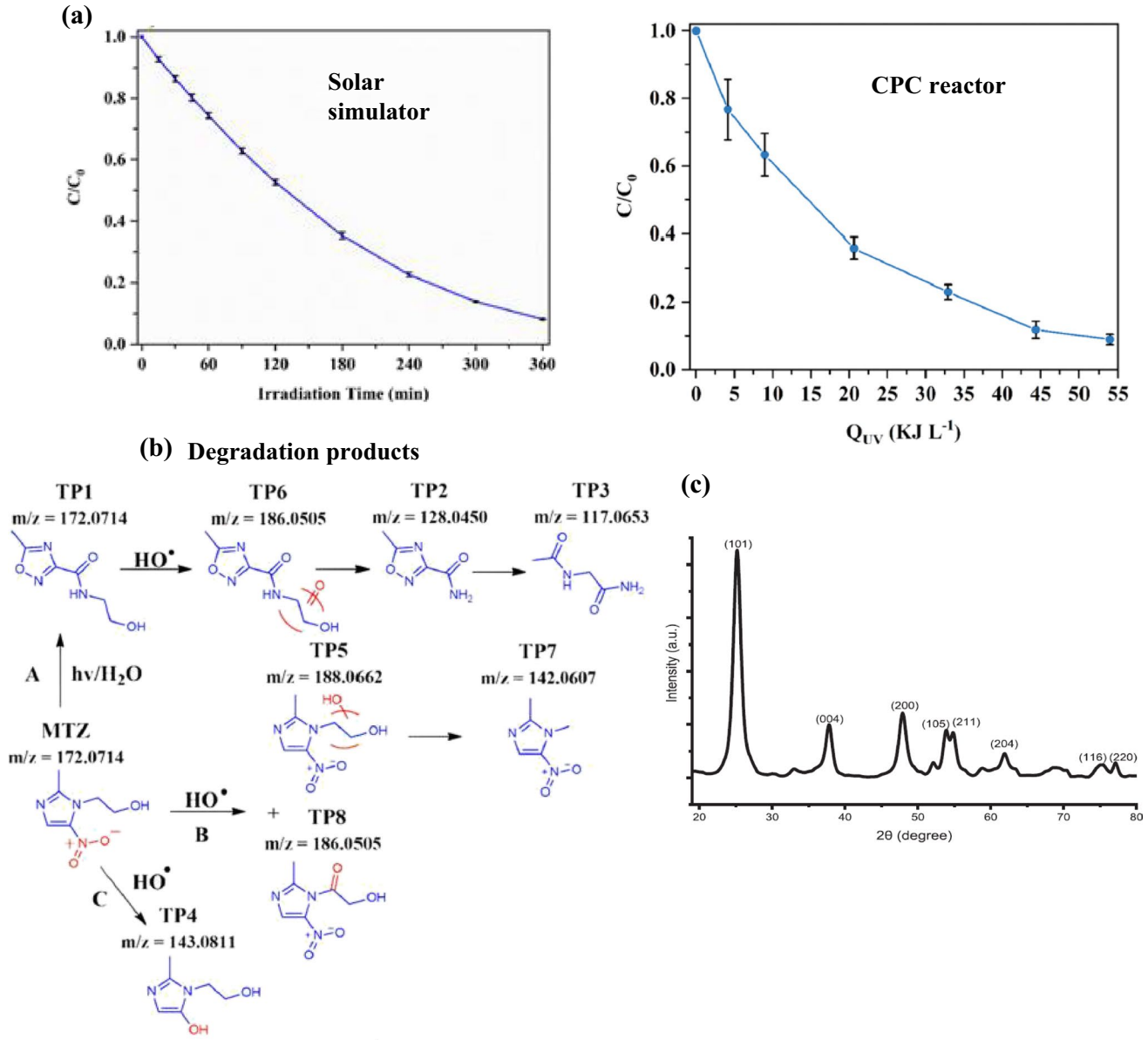

Fig. 10 **a** Comparison of photocatalytic degradation kinetics of metronidazole under solar simulator and CPC, **b** the degradation products metronidazole (Lykos et al. 2023) (Reprinted with permission), **c** XRD of commercial TiO_2 (Akter et al. 2022)

technique still faces several technical barriers limiting its large-scale application. The high rate of charge carriers (electron–hole) recombination and wide bandgap of TiO_2 can limit their photocatalytic effectiveness (Acharya and Parida 2020). Electron–hole recombination occurs, when the electrons and holes generated by light absorption recombine before they can react with contaminants, such as pharmaceuticals. This can reduce the number of active species (reactive oxygen species) available to degrade contaminants (Mishra et al. 2023). The wide band gap of TiO_2 (3.2 eV for anatase) means that it can only absorb light with wavelengths shorter than 380 nm, which limits its responsiveness under visible light and the exploitation of solar energy for photocatalysis (Kader et al. 2022). Visible light accounts for 42–43% of the sun's energy compared to only 3–5% UV irradiation (Mingmongkol et al. 2023). Many strategies have been applied to improve the limitations of TiO_2 with the goal of enhancing its photo absorption in the visible region and photoactivity for the degradation of pharmaceuticals. These include doping of TiO_2 with metals (e.g. Pd, Ag, Au, Cu, Mn, Mo) (Alsaidi et al. 2022; Anucha et al. 2022; Mingmongkol et al. 2023) and non-metals (e.g. N, S, P, C) (Divakaran et al. 2021), combining TiO_2 with other semiconductors (e.g. CdS, ZnO, g-C_3N_4, ZnS, MnO_2) or materials such as carbon

nanostructures (e.g. graphene, carbon nanotubes, carbon dots, activated carbons) (Abdullah et al. 2023; John et al. 2020; Maletić et al. 2019; Noroozi et al. 2022), or immobilizing on various supports such as alumina, glass zeolite and polymeric materials (e.g. chitosan, alginate) (Kumar et al. 2022; Neghi et al. 2019; Sturini et al. 2020) and heterojunction construction (Acharya et al. 2022). Table 5 illustrates different TiO_2-based nanomaterials that have been utilized for the remediation of targeted pharmaceutical compounds.

Although studies have embarked on the application of commercial TiO_2 for the degradation of pharmaceuticals, the importance given to the synthesized TiO_2 also reflects continuous efforts to produce newly engineered TiO_2 materials, with improved optical and surface properties. In addition to that, studies on the coupling of TiO_2, be it commercial or synthesized with other semiconductors, e.g. ZrO_2, Fe_2O_3 and MoS_2, have observed enhanced photocatalytic activity due to the modification in their textural, structural and optical characteristics (Carbuloni et al. 2020; Khasawneh et al. 2021). These include a large surface area, small particle size, a significant proportion of the anatase phase and alterations in the energy band gap (Carbuloni et al. 2020). Thus far, doping of TiO_2 has been performed using various types of metals and non-metals (Table 5). Studies have shown that such an approach has successfully lowered the recombination rate of charge carriers, increased the absorption region to the visible range and increased the specific surface area of the fabricated doped materials. These properties have enhanced the extent of the degradation rate of pharmaceuticals. For example, N-doped TiO_2 coated on fiberglass cloth effectively reduced the indirect band gap from 2.96 eV (in undoped TiO_2) to 2.68 eV (optimum N), leading to enhanced photocatalytic removal efficiencies of carbamazepine, caffeine and ibuprofen by 77%, 88% and 79%, respectively, after 14 h of visible light irradiation (Fig. 11a) (Muangmora et al. 2023). Doping of Mo on TiO_2 also resulted in a visible-light-responsive photocatalyst, which improved removal for paracetamol, due to the availability of surface-active sites and effective charge transfer from TiO_2 to Mo (Kumar et al. 2019). Another study reported that Mn-doped TiO_2 functioned as a visible light-responsive photocatalyst, achieving 95% salbutamol removal within 180 min. The high photocatalytic efficiency was attributed to the 0.2% Mn-doped TiO_2, which exhibited a lower band gap of 2.80 eV compared to the undoped TiO_2 (2.92 eV) (Mingmongkol et al. 2023). Doping TiO_2 with Co ions using the reflux method resulted in a higher photocatalytic degradation efficiency of oxytetracycline hydrochloride compared to the hydrothermal approach (Akel et al. 2020). This improvement was attributed to the reduced band gap of TiO_2, which enhanced its responsiveness to visible light and promoted more effective generation and utilization of electron–hole pairs in the degradation process. Furthermore, effect of environmental-friendly green oxidant like H_2O_2 and peroxymonosulfate on the TiO_2 photocatalytic degradation of pharmaceutical compounds has been also reported (Bosio et al. 2019; Grzegórska et al. 2023; Ncube et al. 2025). For example, UV/TiO_2/H_2O_2 was found to be effective in removing antiretroviral drugs namely emtricitabine, lamivudine and tenofovir. The study revealed that optimum concentration of H_2O_2 is crucial in producing ·OH radicals, which are responsible for enhancing degradation rates, while excessive amounts would capture ·OH radicals leading to low degradation rates (Ncube et al. 2025).

Activated carbon, be it commercial or prepared from raw waste materials, has also been added as a support to TiO_2 to increase its adsorption capacity of pharmaceutical compounds. Remarkable dual properties namely adsorption and photocatalytic activity have been displayed, which have fabricated TiO_2/activated carbon from various precursors, such as nutshell from the Argan tree (El Mouchtari et al. 2020), lignin from lignocellulosic biomass (Peñas-Garzón et al. 2020) and other agricultural wastes such as corncob (Abdullah et al. 2023). The combination of activated carbon from corncob biomass and TiO_2 resulted in an optimum composite material, ACT-4 with a lower band gap energy of 3.05 eV compared to the pure TiO_2 (3.2 eV) as affirmed by UV–Vis diffuse reflectance spectroscopy (Fig. 11b). The highest specific surface area of 1223 m^2/g and photoluminescence spectroscopy results also confirmed that the lowest charge carrier recombination rate in the composite sample reflects high adsorption and mineralization of ceftriaxone to fewer toxic compounds as proposed by the degradation mechanism (Fig. 11c) (Abdullah et al. 2023). Microwave-assisted TiO_2/activated carbon yielded the highest removal of acetaminophen, ibuprofen and antipyrine under a solar simulator compared to two other investigated methods, sol–gel and solvothermal (Peñas-Garzón et al. 2020). Activated carbon, which is known to be low cost, possesses high surface area, good porosity and strong adsorption capacity (Briche et al. 2020; Cunha et al. 2019). Thus, the synthesis of activated carbon/TiO_2 nanomaterials or heterojunction-based photocatalysts could potentially overcome the intrinsic limitations of TiO_2 and produce low-cost materials, with good photocatalytic efficiency in removing various pharmaceutical compounds such as ceftriaxone, sulfamethazine, acetaminophen, ibuprofen and antipyrine.

Besides activated carbon, the inclusion of graphene in the preparation of TiO_2-based materials has been reported to enhance degradation efficacy by promoting charge carrier separation, providing a higher surface area and improving band gap and optical properties (Asadzadeh Patehkhor et al. 2021; Firouzi et al. 2021). Graphene, characterized by sp2-hybridized carbon atoms, serves as an excellent mechanical platform for TiO_2 due to its remarkable capacity for electron transfer and storage, optical transparency and inherently large surface area (Li et al. 2020). The performance of TiO_2/

Table 5 Representative studies of TiO_2-mediated photocatalytic application on pharmaceuticals

TiO_2 or TiO_2-based photocatalysts	Pharmaceutical candidate	Synthesis/preparation method	Type of water matrix	Pollutant conc	Light source or irradiation type	Removal efficiency (%) or degradation rate (min^{-1})	Reference
TiO_2 (synthesized)	Vancomycin	Hydrolysis reaction	Deionized distilled water	15–75 mg/L	UVC (254 nm)	89.5% of removal was achieved at pH 5.1, TiO_2 dosage of 54.9 mg, initial concentration of 58.2 mg/L and reaction time of 36.3 min	Dehghani et al. (2022)
TiO_2/ZnO	Tetracycline	Coprecipitation and ball milling	Not stated	15–60 mg/L	UV lamp (details not provided)	Highest tetracycline degradation was 82% after 165 min using TiO_2:ZnO of 2:1	Zeinali Heris et al. (2023)
TiO_2/ZrO_2	Metformin	Sol–gel	Aqueous solution	10 mg/L	UV mercury lamp (125 W)	5% ZrO_2-TiO_2 (0.5 g/L) produced highest first-order rate constant of 0.0099 min^{-1} of metformin	Carbuloni et al. (2020)
Au/TiO_2	Trimethoprim (TMP) and metronidazole (MNZ)	Photochemical deposition	Distilled water	5, 10 and 20 ppm	High-pressure visible lamps (380–650 nm) and medium-pressure UV lamp (200–290 nm)	TMP: 40 min (complete removal) MNZ: 50 min (complete removal)	Alsaidi et al. (2022)
Mo-TiO_2	Carbamazepine	Sol–gel and sonochemical method	Milli-Q water	5–20 mg/L	UVA lamp (365 nm) and H lamp (visible blue wavelength 300–475 nm)	Mo (15 wt%/TiO_2) of 1 g/L completely removed CBZ within 240 min (unmodified pH)	Anucha et al. (2022)
Ag-TiO_2	Paracetamol	Impregnation	Aqueous solution	10 mg/L	Solar irradiation	1% Ag/TiO_2 resulted in complete degradation of paracetamol after 90 min and 98% TOC removal	Cherif et al. (2023)
g-C_3N_4/TiO_2 and WO_3/TiO_2	Levofloxacin	Hydrothermal	Distilled water	5 mg/L	UV (254 nm) and sunlight	Complete degradation was achieved using both photocatalysts within 30 min under UV light while much longer duration was needed under sunlight	Trifi et al. (2024)

Table 5 (continued)

TiO_2 or TiO_2-based photocatalysts	Pharmaceutical candidate	Synthesis/preparation method	Type of water matrix	Pollutant conc	Light source or irradiation type	Removal efficiency (%) or degradation rate (min^{-1})	Reference
TiO_2-ceramic	Sulfamethoxazole	Glass tube packed with TiO_2 ceramic monolith	Ultrapure water, synthetic mineral water, natural water	5 mg/L	UVA (365 nm)	85.7% removal after 7 h of irradiation at pH 5	Bui et al. (2022)
TiO_2-activated carbon	Acetaminophen, ibuprofen and antipyrine	Solvothermal microwave and sol–gel methods	Deionized water	5 mg/L	Xenon lamp (765–250 W/m^2)	TiO_2-AC synthesized via microwave route yielded highest first-order degradation rate for ibuprofen and antipyrine of 1.40 h^{-1} and 0.25 h^{-1}, respectively. Acetaminophen failed to be degraded after 6 h	Peñas-Garzón et al. (2020)
TiO_2-activated carbon	Diclofenac, carbamazepine sulfamethoxazole	Impregnation method	Ultrapure water	50 mg/L	Xenon lamp (300 W)	AC/TiO_2 9% resulted in complete degradation of diclofenac and while 30% and 50% for carbamazepine and sulfamethoxazole, respectively	El Mouchtari et al. (2020)
PSF/TiO_2/Alg/Ag_3PO_4	Diclofenac and triclosan	Sol–gel and precipitation	Ultrapure water	20 mg/L	LED light (15 W)	Pseudo-first-order rate constants for diclofenac and triclosan was 0.0184 min^{-1} and 0.0520 min^{-1}, respectively, using 10 g/L of beads	Mehmood et al. (2020)
Zeolite/TiO_2	Ibuprofen	Sol–gel and thermal method	Distilled water	50–300 mg/L	UVC lamp (6 W)	Almost complete removal, 99.58% of COD removal was attained using 1 g/L photocatalyst, 0.05 mM H_2O_2 and rection time of 100 min	Farhadi et al. (2021)

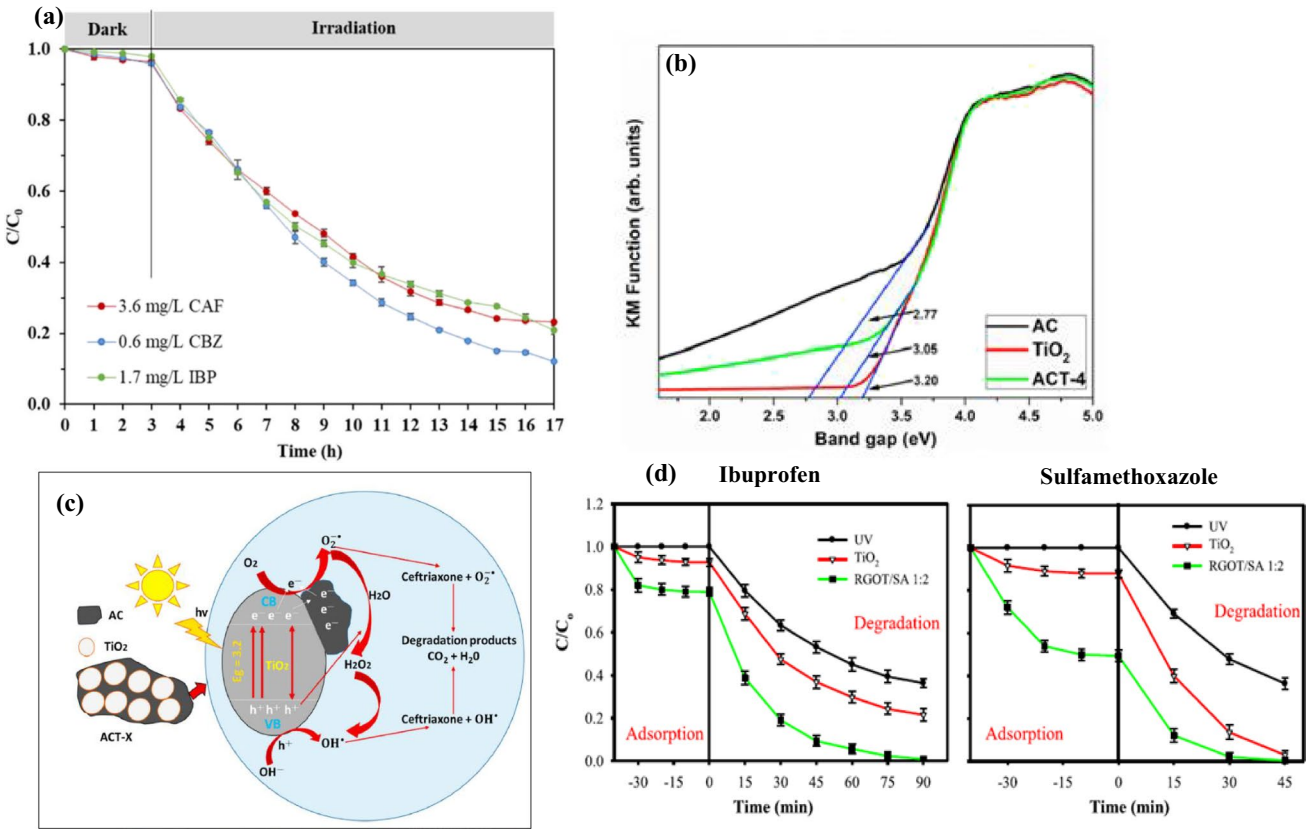

Fig. 11 **a** Degradation of carbamazepine, ibuprofen and caffeine using N-doped TiO_2 coated on fiberglass cloth (Muangmora et al. 2023); **b** comparison of band gap energy between optimum activated carbon-TiO_2 (ACT-4) and its counterparts (Abdullah et al. 2023); **c** mechanism of ceftriaxone degradation using ACT-4 (Abdullah et al. 2023); **d** degradation of ibuprofen and sulfamethoxazole by RGOT/SA aerogel (Nawaz et al. 2020) (Reprinted with permission)

graphene oxide (GO) and TiO_2/reduced graphene oxide (rGO) was validated on acetaminophen (Gomez-Solis et al. 2021) and diclofenac (John et al. 2020), respectively. John et al. (2020) reported that the produced material, TiO_2/rGO, showed a reduced band gap (from 3.25 to 2.75 eV) allowing effective functioning to degrade and mineralize diclofenac under visible light and natural solar light. A recent study by Evgenidou et al. (2023) compared the performance of TiO_2/GO nanocomposite for the removal of the antiviral drug, abacavir, and the findings revealed that the degradation rate was much slower in landfill leachate compared to ultrapure water under simulated solar irradiation, due to the formation of various by-products.

Among various polymers, which have been investigated as TiO_2 supports, chitosan is noted due to being a natural biopolymer, which with highly reactive hydroxyl and amino groups when combined with TiO_2 leads to synergistic adsorption and photocatalytic effects (Bhatt et al. 2023). Studies applied chitosan to prepare TiO_2/biochar/chitosan for the degradation of ciprofloxacin (Afzal et al. 2022) and TiO_2/Fe_2O_3/chitosan nanocomposites for the degradation of ibuprofen (Rajamehala et al. 2023). Both studies revealed that the materials possess excellent sorption and degradation efficiencies owing to their surface properties and can be applied as inexpensive adsorbents for the removal of pharmaceuticals from water. Alginate (Alg)-based gel materials are also known for their characteristics of being non-toxic, low cost, biodegradable and biocompatible (Kanakaraju et al. 2022). As such, beads or hydrogel beads prepared by embedding TiO_2-based nanomaterials have performed well in adsorbing and photo catalytically removing pharmaceutical compounds as demonstrated by Kumar et al. (2022) and Mehmood et al. (2020) through their investigations using S-TiO_2/WS_2/alginate beads and polysulfone/Alg/TiO_2/Ag_3PO_4 beads, respectively. The materials displayed good recyclability compared to powder form and the synergistic effect between the catalyst and Alg resulted in excellent thermal stability and dual adsorption-photocatalytic effects. Likewise, reduced graphene oxide/TiO_2/sodium alginate (RGOT/SA) aerogel also effectively removed ibuprofen and sulfamethoxazole within a 45–90-min treatment time

under UVA irradiation, following pseudo-second-order rate based on adsorption and photodegradation (Fig. 11d) (Nawaz et al. 2020).

In general, the morphological, structural, optical and physical features of the produced TiO_2-based nanomaterials have been linked to the photocatalytic performance in the degradation of pharmaceuticals. The synthesis pathways, conditions and types of precursors used to synthesize TiO_2-based materials greatly controls their properties. Methods like sol–gel (Anucha et al. 2022; Muangmora et al. 2023), hydrothermal (Huang et al. 2021; Kovacic et al. 2020), simple mixing method with the aid of sonicator (Awfa et al. 2019), combined sol–gel and hydrothermal (Payan et al. 2019) and photo deposition method (Gomes et al. 2021) have been applied in related studies. The current trend in TiO_2-based nanomaterials for the remediation of pharmaceuticals also revolves around the construction of hybrid or heterojunction nanostructures to improve photoexcited electron transfer and suppresses charge carrier recombination and enables visible light responsiveness (Acharya and Parida 2020; Zhou et al. 2024). Shajahan and Mohammad (2023) explored the capability of heterojunctions between 2 and 3D nanostructures in the synthesized Co_3O_4/TiO_2/rGO ternary nanocomposite for the removal of ibuprofen and tetracycline, showing efficient transportation of electron–hole pairs and high removal of both pharmaceuticals under visible light. Complete removal of diclofenac was achieved using TiO_2–CdS photocatalysts designed as type-II and direct Z-scheme heterojunction systems (Elangovan et al. 2021). Under visible light irradiation, these systems demonstrated enhanced photocatalytic performance due to improved charge separation, reduced electron–hole recombination and efficient charge transfer.

Studies have also showcased the state-of-the-art modification techniques of TiO_2-based photocatalysts for antibiotic degradation. Firooz et al. (2024) synthesized visible light responsive a TiO_2/natural pyrite S-scheme heterojunction photocatalyst, which led to complete removal of 30 mg/L tetracycline and 84% mineralization (measured as TOC) within 180 min. The excellent removal tetracycline was attributed to the effective utilization of photogenerated electrons in the conduction band of pyrite and holes in the valence band of TiO_2. A recent study reported that $(BiO)_2CO_3/TiO_2$ heterojunction with efficient separation of charge carriers successfully degraded 93.2%, 97.5% and 100% of three antibiotics namely lomefloxacin, ciprofloxacin and norfloxacin, respectively, under simulated sunlight radiation (Gao et al. 2025).

Scaling-up and implementation of AOPs in real environment

Most of the studies discussed in the above sections have been conducted at the laboratory scale with laboratory settings. The scaling up of these processes to an industrial level is crucial to assess their feasibility, efficiency and cost-effectiveness under real-world conditions. Scaling up any chemical process from laboratory scale to industrial scale requires careful consideration of several factors. The approach taken also depends on whether a single AOP or hybrid AOP is being established. For example, for the design of a single ozonation process, the ozonation reactors need to accommodate larger volumes of wastewater, while maintaining efficient contact between ozone and contaminants. This may involve choosing the appropriate reactor type (e.g. bubble column, packed column or membrane reactor) and optimizing its dimensions for the desired flow rate and residence time. Industrial-scale ozonation systems require efficient ozone generation methods capable of producing large quantities of ozone. Ensuring the reliable delivery of ozone to the wastewater stream while minimizing ozone loss is crucial.

Industrial-scale AOP processes require robust control and monitoring systems to maintain optimal operating conditions, including ozone concentration, pH, temperature and contact time. Automation and real-time monitoring can help ensuring consistent performance and identify any deviations or issues promptly. This may involve incorporating renewable energy-powered agitators, pumps or circulation systems to ensure efficient operation. When implementing renewable energy-enabled operations in real environmental conditions, factors such as climate variations, seasonal changes in solar irradiance and geographical location need to be considered. These factors may influence system design, energy production and overall performance.

As the research progresses and shows promising advancement, pilot and commercial scale set up becomes very relevant to ensure the developed materials and processes can be practically implemented in real applications to achieve economic viability. In this context, the materials developed at the laboratory scale should be carefully assessed for their potential to be scaled up. The material stability over a long and continuous reaction process is a major concern in any industrial operation. Ensuring the stable structural integrity and performances under the parameters for real application such as elevated temperatures and high pressure is crucial for their reliable long-term operation. Therefore, it is necessary to evaluate the

long-term stability of the newly developed materials and AOP processes alongside their removal efficiency. In addition, as AOP processes typically involve large amounts of chemicals, therefore using alternative cost-effective raw materials or solvents derived from renewable resources or by-products produced from industries can be beneficial in reducing the overall cost for material production and large-scale operation as well as minimizing the adverse environmental impact. For instance, deep eutectic solvents can serve as a promising alternative for the green synthesis of catalysts used in AOPs.

Conclusion and future outlook

Outlook

The following key areas highlight essential future research directions to enhance the understanding and effectiveness of pharmaceutical wastewater treatment:

Regulatory and Geographical Challenges

- The highest total API concentrations are observed in sub-Saharan Africa, South Asia and South America, where pharmaceutical regulations are less stringent than in developed nations like the USA, UK and Australia. Future research should focus on assessing regulatory gaps and developing standardized monitoring frameworks to mitigate pharmaceutical pollution in these regions.

Expanding Research Beyond Conventional APIs

- Current studies primarily examine the removal of conventional Western pharmaceuticals from WWTPs. Research should expand to include complementary and alternative medicines, which are often overlooked but have the potential to contribute significantly to pharmaceutical contamination.

Risk Assessment for Mixed Pharmaceutical Systems

- Pharmaceuticals typically exist in the environment as complex mixtures rather than isolated compounds. Future studies should develop risk assessment models that account for interactions between multiple pharmaceuticals, their transformation products and metabolites.

Optimization of Removal Efficiency and Hazard Reduction

- Studies should focus on analyzing by-products and degradation pathways to optimize removal efficiency while minimizing hazardous by-products.
- Real-time monitoring techniques should be employed to enhance the accuracy of degradation studies.
- Integration of AOPs with membrane technologies should be explored to improve pharmaceutical removal efficiency.

Resource Recovery from Pharmaceutical Wastewater

- Research should explore whether valuable metals or other useful compounds can be recovered from degradation by-products.
- This recovery aligns with the broader goal of sustainable and resource-efficient wastewater treatment.

Enhancing TiO_2-Based Photocatalysts for Pharmaceutical Removal

- Comprehensive studies should investigate how different molecular structures and chemical properties influence interactions with TiO_2-based photocatalysts.
- Long-term stability and environmental impact assessments are needed to determine the feasibility of scaling up these photocatalysts for real-world applications.

Computational and AI-Driven Approaches

- A multidisciplinary approach should be emphasized by integrating experimental studies with computational methods such as machine learning (ML) and AI-based predictive modelling.
- Research should focus on optimizing ML algorithms for classification, pattern recognition and predictive modelling of pharmaceutical-TiO_2 interactions.
- Predictive models should be developed to facilitate efficient material screening and prioritize potential photocatalyst candidates, reducing reliance on extensive experimental screening.

Conclusion

This review has clearly demonstrated that there have been advances in the application of AOPs for the removal of pharmaceuticals from wastewater in the last 5 years (Fig. 12). Pharmaceuticals containing APIs, while promoting human and animal health, present a challenge due to their persistence in the environment, with the extent of the problem determined not only by their physicochemical properties but their ability to transform to by-products. Pharmaceuticals from various sources have become pollutants in our environment, primarily entering water systems through wastewater treatment plants but also from farms, households, industries and hospitals. Their complex nature and coexistence with other pollutants often leads to incomplete removal in these treatment plants, resulting in their release into the environment. Pharmaceuticals have been found to cause a range of

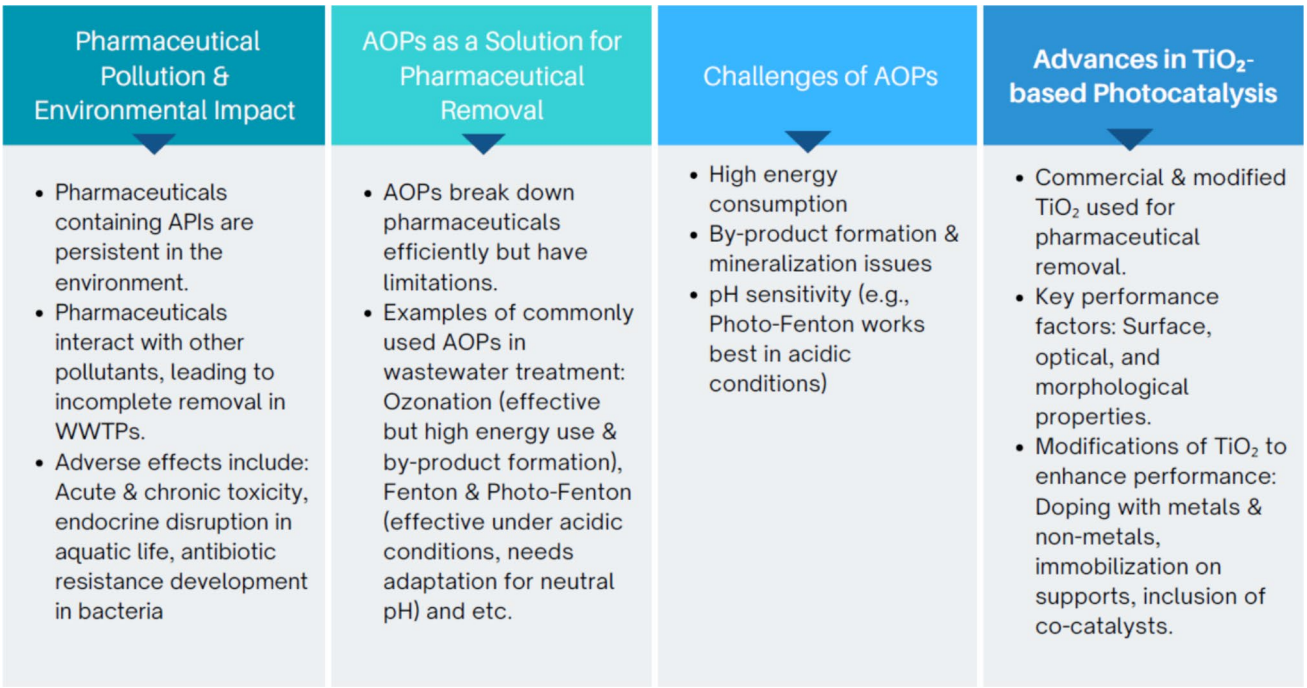

Fig. 12 Important findings derived from the review

adverse effects, from those which are acute and chronic to endocrine disruption in aquatic life, and the development of antibiotic resistance in bacteria. The complex behaviour of pharmaceuticals, influenced by various factors, emphasizes the necessity for further research on their efficient removal from the environment. It is important to gain a deeper understanding of how pharmaceuticals behave in different environmental contexts and to develop effective strategies to mitigate their potential risks to both the environment and human health. To address this issue, various methods have been developed for pharmaceuticals removal, with AOPs continuing to show promise due to their ability to efficiently break down these pollutants.

Recent studies have revealed the continued application of multiple AOPs in pharmaceutical wastewater treatment, including ozonation, Fenton and photo-Fenton reactions, electrochemical methods, photocatalysis, photochemical processes, sonolysis and hybrid approaches. Despite their effectiveness in removing numerous APIs, challenges persist within the utilization of these technologies. For example, ozonation has the potential for treating pharmaceutical wastewater, but challenges like by-product formation and high energy use remain, necessitating further research for more effective and environmentally friendly removal of these pollutants. Photo-Fenton processes are traditionally effective under acidic conditions (pH 2–3), making it a challenge to adapt this method for near-neutral pH levels, which would enhance its attractiveness. In addition to the established UV and UV/H_2O_2 processes, the UV/PAA method is gaining prominence for pharmaceutical degradation. However, it is essential to acknowledge that while these processes can effectively break down parent compounds, addressing issues related to mineralization and the presence of degradation by-products continues to require improvement. Hybrid processes, in contrast to the single treatment options, have yielded outstanding pharmaceutical removal and degradation of by-products such as the combination of TiO_2 photocatalysis and ozonation. In the case of TiO_2 photocatalysis, both commercial and modified TiO_2 can be applied for the eradication of pharmaceutical contaminants in water. The surface, optical and morphological properties are crucial in determining the photocatalytic performance of the engineering TiO_2-based nanomaterials. Modifications of pristine TiO_2 have been undertaken via doping with metals and non-metals, immobilization of supports and inclusion of cocatalysts.

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Declarations

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