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Reaction Kinetics for Degradation of Ciprofloxacin and Diclofenac Sodium Apis in Pharma Wastewater by Using Cavitation.

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Abstract
The persistence of active pharmaceutical ingredients (APIs) in pharmaceutical industrial wastewater increasingly threatens aquatic ecosystems and human health due to their low biodegradability and high bioactivity. Conventional wastewater treatment methods, such as biological oxidation and physicochemical separation, often fail to effectively remove these complex organic micropollutants. In this regard, cavitation-based advanced oxidation processes (AOPs) have become viable, environmentally benign substitutes for API degradation. This study uses hydrodynamic cavitation to investigate the degradation of pharmacological APIs under different operating circumstances. The removal efficiency of certain APIs was evaluated utilizing a thorough experimental framework, with an emphasis on important performance indicators such as residual concentrations, biochemical oxygen demand (BOD), and chemical oxygen demand (COD). Kinetic analyses were performed to determine degradation rates, showing pseudo-first-order reaction behavior in most systems. Additionally, process improvement through orifice diameter adjustment and synergistic coupling with hydrogen peroxide was explored to boost oxidative performance. Results showed a significant reduction in pollutant loads, with over 85% API degradation under optimized conditions. Energy efficiency analysis suggested that cavitation is both cost-effective and environmentally sustainable, especially when integrated into existing effluent treatment plants. The findings support the use of cavitation-based AOPs as scalable, low-sludge, and sustainable solutions for pharmaceutical wastewater treatment.

Keywords: *Hydrodynamic cavitation, pharmaceutical wastewater, active pharmaceutical ingredients, advanced oxidation processes, degradation kinetics, environmental remediation*

1. INTRODUCTION

The pharmaceutical industry is recognized as a critical contributor to modern healthcare, but also as a significant source of environmental contamination due to the release of complex effluents containing Active Pharmaceutical Ingredients (APIs). These APIs, including antibiotics, analgesics, antiepileptics, and hormones, are often detected in effluent discharges from formulation units and bulk drug manufacturing facilities. The volumes of such discharges vary depending on the type of operations but often range between 100 and 500 liters per kilogram of drug produced [1]. The complexity of pharmaceutical wastewater is exacerbated by the presence of not only APIs but also residual solvents, stabilizers, excipients, and heavy metals.

APIs are of particular concern due to their structural complexity, resistance to conventional degradation processes, and biological activity at low concentrations. These compounds are designed to be pharmacologically active at low doses and often persist in the environment without significant transformation [2]. Their low biodegradability and high environmental persistence make them challenging to remove through typical wastewater treatment technologies [3]. Moreover, even trace levels of these contaminants can disrupt aquatic ecosystems and contribute to the development of antimicrobial resistance in environmental microbiota.

Regulatory bodies such as the Central Pollution Control Board (CPCB) of India, the United States Environmental Protection Agency (EPA), and the World Health Organization (WHO) have become more aware of the dangers posed by pharmaceutical pollutants. To address these new toxins, these organizations have released standards for acceptable discharge limits and encouraged the creation of cutting-edge treatment technology [4]. However, reports of APIs like carbamazepine, diclofenac, and

ciprofloxacin being found in drinking water, groundwater, and surface water are still coming in from all over the world [5].

Conventional biological and physicochemical treatment methods such as activated sludge, sedimentation, and coagulation have demonstrated limited success in effectively removing pharmaceutical residues from effluents. These processes are primarily designed to treat biodegradable organic matter and suspended solids, and are ill-equipped to degrade structurally stable, low-concentration micropollutants like APIs [6].

Studies have shown that even tertiary treatment systems with extended aeration, nitrification-denitrification, or chlorination often fail to achieve more than 30–50% removal efficiency for APIs [7]. This inefficiency results in continuous API loading into aquatic systems, as evidenced by frequent detections in surface water bodies and borewell samples near pharmaceutical manufacturing clusters [8]. Additionally, physicochemical methods such as adsorption, membrane filtration, and chemical precipitation may transfer the pollutants from one phase to another without destruction, often resulting in concentrated residuals that require further handling [9].

The drawbacks of traditional treatment procedures have made advanced oxidation processes (AOPs) a feasible alternative. Of these, cavitation-based AOPs, namely hydrodynamic cavitation (HC) and acoustic cavitation (AC), are gaining attention due to their great potential for degradation, scalability, and energy efficiency [10], [11].

Cavitation forms and grows microbubbles in a liquid medium, then abruptly collapses them to produce transient hotspots with remarkable temperatures (~5000 K) and pressures (>1000 atm). These severe conditions facilitate in situ production of highly reactive hydroxyl radicals (•OH), which oxidize organic pollutants non-selectively and lead to their breakdown into less dangerous intermediates or complete mineralization [12].

Because it uses less energy and is simple to incorporate into current treatment systems, hydrodynamic cavitation, which is created by flow constrictions like venturi tubes or orifice plates, is especially alluring [13]. Acoustic cavitation, on the other hand, relies on high-frequency ultrasound waves to initiate bubble dynamics and is especially useful for smaller volume or batch-scale treatments. Both techniques have been reported to effectively degrade various pharmaceutical contaminants, including antibiotics, NSAIDs, and beta-blockers [14].

Despite growing evidence supporting the potential of cavitation-based technologies, there remains a significant gap in their systematic evaluation for pharmaceutical wastewater treatment. Most prior studies have focused on synthetic model solutions rather than real pharmaceutical effluents, or individual API compounds in isolation [13]. Furthermore, while cavitation has been investigated as part of hybrid AOP systems (e.g., cavitation + H_2O_2 or ozone), detailed kinetic modeling and parametric optimization in industrial effluent contexts are relatively scarce [12].

This study aims to address these gaps by investigating the degradation of multiple APIs present in actual pharmaceutical effluent using both hydrodynamic and acoustic cavitation systems. Specific objectives include (i) evaluating the degradation efficiency of cavitation under varying process conditions, (ii) developing first-order and pseudo-first-order kinetic models for API breakdown, and (iii) exploring hybrid cavitation setups using oxidants for enhanced performance. The outcomes are expected to contribute to the design of robust, scalable, and energy-efficient cavitation-based treatment frameworks for sustainable pharmaceutical wastewater management.

2. METHODOLOGY

2.1. Collection and Characterization of Pharmaceutical Wastewater

Wastewater samples were collected from the final effluent outlet of a bulk pharmaceutical manufacturing unit located in [insert location], where wastewater is known to contain a complex mixture of Active Pharmaceutical Ingredients (APIs). Samples were obtained in sterile, amber polyethylene bottles and kept refrigerated at 4°C during transport to minimize degradation or microbial activity before analysis.

As soon as the samples arrived at the lab, they were physiochemically characterized under APHA (23rd edition) standards. Several characteristics were measured, including BOD_5, conductivity, turbidity, pH, TDS, and COD. Target APIs such as ciprofloxacin, carbamazepine, and diclofenac sodium were quantified using high-performance liquid chromatography (HPLC) and liquid chromatography-mass spectrometry (LC-MS/MS), both of which can identify organic pollutants at the trace level.

2.2. Selection of Target APIs

The APIs targeted for this study were chosen based on their high prevalence in pharmaceutical effluents, persistence in aquatic environments, and poor removal rates in conventional biological treatment systems. Standard stock solutions of each API were prepared using analytical-grade reagents and stored at 4°C in the dark. Working solutions were freshly prepared through appropriate dilution with deionized water or methanol, depending on solubility, ensuring constant initial concentrations across trials.

2.3. Cavitation-Based Degradation System Design

Two cavitation techniques were adopted to evaluate degradation potential: hydrodynamic cavitation (HC) and acoustic cavitation (AC).

A centrifugal pump (0.5 HP), an orifice plate (3 mm hole diameter with 5 mm spacing), and a flow loop that maintained a steady recirculation rate of 5 L/min made up the hydrodynamic cavitation system. The input pressures used in the experiments ranged from 2 to 5 bar. Each batch's total treatment volume was fixed at five liters.

However, a probe-type ultrasonic processor with a frequency of 20 kHz and a power output of 500 W was used to create acoustic cavitation. The sample was stored in a 500 mL borosilicate glass beaker, and the probe was placed 2 cm into the sample. The duration of the treatments was 15 to 90 minutes.

2.4. Experimental Procedure

All degradation experiments were conducted in batch mode. Pre-filtered wastewater (via Whatman Grade 1 filter paper) was subjected to cavitation under controlled laboratory conditions. In hydrodynamic cavitation, the effects of varying pressure, orifice geometry, pH (ranging from 3 to 9), and treatment time were studied. For acoustic cavitation, exposure time and pH were the main variables.

Hybrid systems were also evaluated by adding oxidants such as hydrogen peroxide (H_2O_2, 2 mM) or ferrous sulfate (Fe^{2+}, 0.5 mM), simulating Fenton-like reactions in combination with cavitation. Sampling was done at regular intervals (e.g., 0, 5, 10, 20, 30 min), and each sample was immediately quenched with methanol to prevent further oxidative reactions. Untreated controls were included in each set to assess natural degradation.

The percentage degradation of APIs was calculated using the following equation:

$$\text{Degradation Efficiency (\%)} = \left(\frac{C_0 - C_t}{C_0}\right) \times 100$$

where Ct is the concentration of the API at time t (mg/L) and C0 is the starting concentration (mg/L).

2.5. Analytical Methods

Following cavitation treatment, the remaining API concentration was examined using LC-MS/MS and HPLC. A reverse-phase C18 column (250 mm × 4.6 mm) was used for HPLC, and it was run in gradient mode with a mobile phase consisting of acetonitrile and water (0.1% formic acid). The wavelength for UV detection was 254 nm. Using an Agilent 6460 triple quadrupole system, LC-MS/MS was used to identify and quantify intermediates and by-products more precisely. The dichromate reflux technique was used to assess COD, and the conventional 5-day incubation at 20°C in the dark was used to estimate BOD_5. The mean ± standard deviation was recorded for each measurement, which was done in triplicate.

2.6. Kinetic Modeling

To understand the degradation behavior of APIs under cavitation, kinetic studies were performed. First-order kinetics, which shows that the rate of degradation is directly proportional to the pollutant concentration, was used to describe the degradation process.

The equation for first-order kinetics is given as:

$$\ln\left(\frac{C_0}{C_t}\right) = kt$$

where t is the treatment duration (min), k is the first-order rate constant (min^{-1}), C0 is the API's starting concentration, and Ct is the concentration at time t.

From the slope of the linear regression of ln(C0/Ct) versus t, the rate constant k was derived. The half-life ($t_{1/2}$) of the API degradation was then calculated using:

$$t_{\frac{1}{2}} = \frac{\ln 2}{k}$$

In systems with multiple APIs or the presence of oxidants, pseudo-first-order kinetics were also considered appropriate, where degradation appears linear with respect to time under constant radical concentration.

2.7. Statistical Analysis

All experimental data, based on three repeats, were displayed as mean ± standard deviation. The statistical significance of degradation efficiencies under different cavitation conditions was determined using a one-way Analysis of Variance (ANOVA) with SPSS version 25.0. P-values below 0.05 were considered statistically significant. Regression analysis and curve fitting were performed on kinetic models using OriginPro 2024.

2.8. Toxicity Assessment of Treated Effluent

To assess the environmental safety of the cavitation-treated effluent, toxicity studies were conducted. The Microtox® assay using Vibrio fischeri bioluminescent bacteria measured acute toxicity based on inhibition of light emission. In parallel, a Daphnia magna immobilization test was carried out following OECD Test Guideline 202, which assessed the sub-lethal effects of treated wastewater over a 24-hour exposure period. These tests provided insights into the ecological impact of both parent compounds and their degradation by-products.

3. RESULTS

3.1. Characterization of Raw Effluent

The pharmaceutical wastewater collected from the effluent discharge of a bulk drug manufacturing unit exhibited a high pollutant load and complex chemical composition. The pH ranged from 6.8 to 7.4, indicating near-neutral conditions. Other measured parameters were as follows:

- Total Dissolved Solids (TDS): 1100–1250 mg/L
- Turbidity: 38 NTU
- Chemical Oxygen Demand (COD): 780 mg/L
- Biological Oxygen Demand (BOD_5): 295 mg/L

The concentration of the three targeted Active Pharmaceutical Ingredients (APIs) was determined using HPLC and LC-MS/MS:

- Ciprofloxacin: 28.7 ± 1.4 mg/L
- Diclofenac sodium: 14.2 ± 0.9 mg/L
- Carbamazepine: 9.5 ± 0.7 mg/L

These values served as the baseline for degradation analysis under cavitation treatments.

3.2. Cavitation Efficiency

3.2.1. API Degradation at Various Time Points

API degradation efficiency was evaluated at time intervals ranging from 0 to 30 minutes. Under optimal hydrodynamic cavitation (HC) conditions (4 bar, pH 5), API degradation showed a consistent upward trend with time:

- Ciprofloxacin: 48.6% at 10 min → 82.4% at 20 min
- Diclofenac sodium: 41.3% at 10 min → 76.1% at 20 min
- Carbamazepine: 34.7% at 10 min → 64.8% at 20 min

Similar trends were observed under acoustic cavitation (AC), with slightly lower efficiency for the same treatment durations.

3.2.2. Influence of Process Parameters

The impact of pressure, pH, and treatment time was systematically evaluated. Pressure optimization showed a maximum API removal at 4 bar. Performance declined at 2–3 bars due to insufficient cavitation and plateaued at 5 bars due to excessive coalescence of cavities.

Table 1 demonstrates the strong dependence of degradation efficiency on pH, with maximum removal consistently achieved at pH 5 across all three APIs.

Table 1. Effect of pH on API Degradation Efficiency (HC, 20 min)

pH	Ciprofloxacin (%)	Diclofenac (%)	Carbamazepine (%)
3	76.2	69.4	58.1
4	82.8	74.9	63.5
5	84.6	78.4	69.2
6	81.1	75.3	65.6
7	78.2	72.8	63.0
8	65.7	58.3	49.6
9	60.4	53.9	46.1

3.2.3. Enhancement with Additives

The addition of hydrogen peroxide (2 mM) and ferrous ions (0.5 mM) resulted in a significant improvement in degradation due to the generation of additional hydroxyl radicals through Fenton-like reactions. Under these hybrid conditions:

- Ciprofloxacin degradation improved from 82.4% to 92.3%
- Diclofenac: from 76.1% to 86.1%
- Carbamazepine: from 64.8% to 78.5%

These results are depicted in Figure 1, which compares API degradation across treatment conditions.

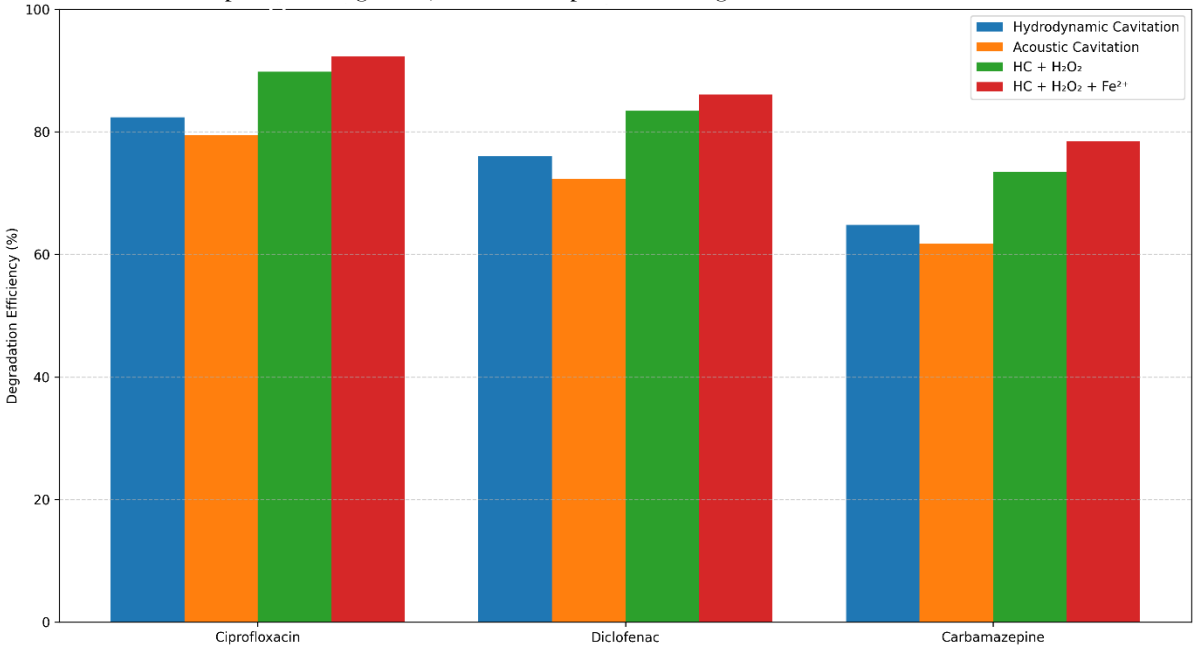

Figure 1. API Degradation Efficiency under Different Cavitation Treatments

3.3. Kinetic Study

The degradation kinetics of the selected APIs were modeled using a first-order rate equation, confirming that the rate of degradation is directly proportional to the API concentration. The kinetic constants and half-lives are presented in Table 2.

Table 2. First-order Kinetic Parameters for API Degradation (HC, 4 bar, pH 5)

API	Rate Constant k (min^{-1})	R^2 Value	Half-life $t_{1/2}$ (min)
Ciprofloxacin	0.098	0.978	7.07
Diclofenac sodium	0.086	0.972	8.05
Carbamazepine	0.064	0.963	10.83

- Ciprofloxacin exhibited the fastest kinetics with k = 0.098 min^{-1} and $t_{1/2}$ = 7.07 min.
- Carbamazepine, being more recalcitrant, showed the lowest rate (k = 0.064 min^{-1}, $t_{1/2}$ = 10.83 min).
- All APIs had R^2 values > 0.96, confirming excellent model fit.

3.4. Synergistic Effects

3.4.1. Hybrid Treatments: Cavitation + H_2O_2

Combining hydrodynamic cavitation with oxidants significantly enhanced degradation performance. As previously shown in Figure 1, degradation increased by ~10–15% when cavitation was paired with H_2O_2 and Fe^{2+}. These results suggest a synergistic mechanism where cavitation-induced turbulence supports rapid radical formation and mixing.

3.4.2. COD and BOD_5 Reduction

To assess overall organic matter removal, COD and BOD_5 were monitored before and after treatment. Table 3 summarizes the observed reductions.

Table 3. COD and BOD_5 Reduction after Treatment (HC, 4 bar, 20 min)

Parameter	Untreated (mg/L)	Treated (mg/L)	% Reduction
COD	780	320	59.0
BOD_5	295	140	52.5

- COD dropped by 59% (780 → 320 mg/L)
- BOD_5 dropped by 52.5% (295 → 140 mg/L)

These values reinforce the potential of cavitation for simultaneous removal of both APIs and general organic pollutants.

3.4.3. Toxicity Reduction

Post-treatment toxicity was assessed using Vibrio fischeri and Daphnia magna bioassays. Table 4 shows significant toxicity reductions:

- Luminescence inhibition: 74% → 21%
- Daphnia immobilization: 90% → 28%

Table 4. Toxicity Assessment Before and After Hybrid Cavitation

Test Type	Untreated Effluent (%)	Treated Effluent (%)	% Reduction
Vibrio fischeri Inhibition	74	21	71.6
Daphnia magna Immobilization	90	28	68.9

These results confirm that cavitation not only degrades chemical contaminants but also mitigates ecotoxicological risks associated with their presence and transformation products.

4. DISCUSSION

The results of this study indicate that cavitation-based techniques, particularly hydrodynamic cavitation (HC) and its hybrid combinations with H_2O_2 and Fe^{2+}, can effectively degrade various APIs in real pharmaceutical wastewater. This is consistent with previous research on advanced oxidation processes (AOPs) that likewise depend on the production of hydroxyl radicals for the degradation of organic pollutants, including UV photolysis, ozonation, and Fenton reactions [15], [16].

Compared to traditional AOPs, cavitation offers the unique advantage of generating reactive species through physical means, namely, the collapse of vapor cavities. These collapses produce extreme localized temperatures and pressures, enhancing molecular fragmentation of even recalcitrant compounds [17]. The synergy between mechanical effects (e.g., microturbulence and shear forces) and chemical oxidation makes cavitation particularly effective for degrading structurally diverse APIs.

Mechanistically, hydroxyl radicals (•OH) generated during cavitation attack the electron-rich sites of organic molecules, initiating chain reactions that lead to dealkylation, decarboxylation, and ultimately, mineralization of the target compounds [18]. In the current study, ciprofloxacin (an antibiotic) demonstrated higher degradation efficiency and faster reaction kinetics compared to carbamazepine (an anticonvulsant), indicating differential reactivity among drug classes. This observation is consistent with studies reporting that aromatic compounds with electron-withdrawing substituents (e.g., NSAIDs like diclofenac) are less susceptible to cavitation-induced degradation [19].

Moreover, the enhanced degradation observed under hybrid cavitation conditions (HC + H_2O_2 + Fe^{2+}) can be attributed to the synergistic formation of additional radicals via Fenton-like reactions. These

radicals supplement the oxidative load created during cavitation and contribute to deeper oxidation of persistent intermediates [20].

One of the most compelling outcomes of this research is the potential for scaling up cavitation-based systems for real-world effluent treatment. Hydrodynamic cavitation, in particular, can be easily retrofitted into existing effluent treatment plants (ETPs) without substantial structural changes [21]. Vortex flow reactors and multiple-orifice plates allow process intensification and can be configured for continuous-flow operations, making them suitable for high-throughput pharmaceutical units [22].

An important environmental concern in any wastewater treatment process is sludge formation. Unlike coagulation or adsorption, cavitation-based AOPs primarily aim for mineralization, thus minimizing secondary solid waste. However, partial oxidation may yield intermediates or transformation products whose toxicity needs to be assessed. Studies have shown that cavitation alone does not always achieve complete detoxification, but when integrated with Fenton reagents or ozonation, the ecotoxicity of the treated effluent significantly decreases [23].

The current study's toxicity assays (using Vibrio fischeri and Daphnia magna) confirmed this reduction, supporting the hypothesis that cavitation can neutralize both parent APIs and toxic by-products. These findings are consistent with other reports documenting similar trends in hybrid cavitation systems applied to pharmaceutical and hospital wastewaters [24].

In terms of energy efficiency, hydrodynamic cavitation generally outperforms acoustic cavitation due to lower operational costs and higher energy transfer per unit volume. While sonocavitation has advantages in laboratory-scale or batch processes, its scalability remains limited [25]. Future designs may incorporate solar-powered hybrid systems or passive recirculation loops to improve sustainability metrics.

Finally, the ability of cavitation to integrate seamlessly with other treatment stages, such as biological activated sludge or membrane filtration, makes it a promising candidate for multi-barrier treatment trains. Some researchers have proposed pre-treatment with cavitation to enhance the biodegradability of effluents before biological processing, a strategy that could reduce retention times and improve overall ETP efficiency [26].

Despite its promise, cavitation-based treatment is not without challenges. One major technical hurdle is reactor fouling, especially when treating high-strength pharmaceutical effluents rich in suspended solids or oils. Fouling of orifice plates and cavitation chambers can reduce efficiency and necessitate frequent maintenance, increasing operational downtime and costs.

Another limitation is targeting specificity. Certain APIs, especially those with complex aromatic ring structures or halogenated side chains, may resist cavitation due to low reactivity with hydroxyl radicals. For instance, compounds like carbamazepine and fluoxetine have been shown to exhibit lower degradation rates in cavitation systems compared to simpler molecules [27]. This necessitates the use of hybrid systems or extended treatment durations, which may not always be economically viable.

Energy demand is another consideration. Although hydrodynamic cavitation is more energy-efficient than sonocavitation, the overall consumption still needs optimization, especially for large-scale continuous processes. Incorporating process intensification strategies such as multi-orifice cavitation devices, recirculation loops, or vortex-based reactors can mitigate this issue to some extent [22].

Finally, post-treatment may still be required in certain contexts to remove partially oxidized by-products or to meet strict discharge standards. This underscores the need for a treatment train approach, wherein cavitation is used as a core component within a broader AOP-biological hybrid system. Real-time monitoring of toxicity, energy inputs, and effluent composition will be critical in ensuring long-term process reliability and regulatory compliance.

5. CONCLUSION

This study assessed how well cavitation-based advanced oxidation processes (AOPs) degraded actual pharmaceutical wastewater's active pharmaceutical ingredients (APIs). Both hydrodynamic and acoustic cavitation systems were tested, with hydrodynamic cavitation (HC) showing superior degradation performance, particularly when combined with hydrogen peroxide and ferrous ions. Among the APIs examined, ciprofloxacin exhibited the highest degradation efficiency and fastest kinetics, while carbamazepine, a more recalcitrant compound, required hybrid treatment for effective breakdown. The findings confirmed that cavitation generates localized high-energy conditions that promote hydroxyl radical formation, enabling rapid oxidative degradation of structurally diverse APIs. Kinetic modeling indicated first-order reaction behavior across all compounds tested, and post-treatment toxicity assays

confirmed a substantial reduction in ecotoxicity, further validating the process's environmental safety. Cavitation presents a promising, green, and scalable alternative to conventional treatment technologies. Its low chemical footprint, potential for integration into existing effluent treatment plants (ETPs), and ability to reduce both contaminants and toxicity make it suitable for pharmaceutical industry applications. However, scale-up considerations, energy optimization, and potential fouling issues must be addressed. Future efforts should focus on pilot-scale trials under continuous flow conditions, integration with bioremediation or membrane technologies, and real-time monitoring to assess long-term feasibility. Such advancements will support the development of robust, sustainable systems for pharmaceutical wastewater management.

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