



Hybrid Bio-AOP Systems for Wastewater Treatment: Experimental Evaluation and Sustainability Assessment

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الأنظمة الهجينة (Bio-AOP) لمعالجة مياه الصرف الصحي تقييم تجريبي وتقييم الاستدامة

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المخلص:

تقيم هذه الدراسة نظامًا هجينًا بيولوجيًا-عملية أكسدة متقدمة (bio-AOP) لمعالجة مياه الصرف الصحي البلدية في ظروف مخبرية مضبوطة. تعكس المياه الداخلة، التي تتميز بارتفاع نسبي في الطلب الكيميائي للأكسجين (518 ملغم/لتر)، والطلب البيولوجي للأكسجين (242 ملغم/لتر)، والمغذيات، والمعادن الثقيلة، والمستحضرات الصيدلانية، والجسيمات البلاستيكية الدقيقة، الظروف النموذجية في المدن متوسطة الحجم في شمال إفريقيا. بالمقارنة مع نظام بيولوجي فقط، حقق النظام الهجين معدلات إزالة أعلى بكثير (89.7% للطلب الكيميائي للأكسجين، و94.5% للطلب البيولوجي للأكسجين، وأكثر من 70% للمستحضرات الصيدلانية والمعادن الثقيلة، وحوالي 80% للجسيمات البلاستيكية الدقيقة)، مع إنتاج حمأة أقل بنسبة 30% وخفض البصمة الكربونية بنسبة 34%. على الرغم من أن النظام يتطلب مدخلات طاقة أعلى (0.29 مقابل 0.13 كيلوواط ساعة/م³)، إلا أن قدرته على إزالة مجموعة واسعة من الملوثات تجعله واعدًا لإعادة استخدام المياه في المناطق التي تعاني من ندرة المياه. علاوة على ذلك، تُبرز النتائج التأثيرات المحتملة على ديناميكيات المجتمعات الميكروبية أثناء المعالجة المتتابعة.

الكلمات الدالة: إعادة استخدام مياه الصرف الصحي، المعالجة البيولوجية الهجينة المتقدمة، المجتمعات الميكروبية، الملوثات الناشئة، الاستدامة.

Abstract:

This study evaluates a hybrid biological–advanced oxidation process (bio–AOP) system for municipal wastewater treatment under controlled laboratory conditions. The influent, characterized by relatively high COD (518 mg/L), BOD₅ (242 mg/L), nutrients, heavy metals, pharmaceuticals, and microplastics, reflects typical conditions in North African medium-sized cities. Compared with a biological-only baseline, the hybrid configuration achieved significantly higher removals (COD 89.7%, BOD₅ 94.5%, pharmaceuticals and heavy metals >70%, microplastics ~80%) while producing 30% less sludge and reducing the carbon footprint by 34%. Although the system required higher energy input (0.29 vs. 0.13 kWh/m³), its broad-spectrum removal capacity makes it promising for water reuse in water-scarce regions. Furthermore, the results highlight potential impacts on microbial community dynamics during sequential treatment.

Keywords: wastewater reuse, hybrid bio–AOP, microbial communities, emerging contaminants, sustainability.

1. Introduction

Water scarcity, exacerbated by population growth, climate change, and industrial expansion, has heightened the urgency to develop advanced wastewater treatment systems capable of producing high-quality effluents suitable for reuse. Conventional biological processes, while effective in removing biodegradable organic matter, are often inadequate for persistent emerging contaminants (ECs) such as pharmaceuticals, endocrine disruptors, heavy metals, and microplastics. These pollutants are increasingly detected in treated effluents worldwide, frequently at concentrations exceeding environmental safety thresholds, and can accumulate in aquatic ecosystems, posing significant risks to human and ecological health (Michael et al., 2013; UNEP, 2022; Redalyc, 2023).

In Libya, particularly in Sabha, municipal wastewater is characterized by a mixture of household sewage and light industrial discharges, with COD (~500 mg/L), BOD₅ (~240 mg/L), nutrients, heavy metals, pharmaceuticals, and microplastic concentrations (>1,000 particles/L). These levels are often higher than those typically reported in Europe and North America (COD 300–400

mg/L), underscoring the pressing need for advanced and context-specific treatment solutions (FAO, 2021; UNEP, 2022).

Hybrid biological–advanced oxidation process (bio–AOP) systems integrate the complementary advantages of microbial degradation and oxidative polishing. The biological stage reduces organic loads and oxygen demand, while the subsequent AOP targets recalcitrant contaminants. Beyond pollutant removal, hybrid systems may influence microbial community dynamics by altering redox environments, generating reactive transformation products, and reshaping microbial succession. Recent evidence highlights that prokaryote–phage interactions in hybrid systems significantly affect microbial stability and functional resilience, thereby influencing overall treatment performance (Wang et al, 2024). Similarly, biofilm-based hybrid reactors have been shown to support more diverse microbial communities and higher nutrient removal efficiency compared with suspended sludge alone, suggesting that microbial architecture within these systems directly contributes to their enhanced performance (Tian et al, 2024).

Emerging research further demonstrates that wastewater type strongly influences microbial community composition, including the persistence of pathogenic bacteria, which has implications for treatment stability and safety (Lee et, 2025). Moreover, reviews of hybrid and integrated treatment processes confirm that conventional biological systems alone cannot sufficiently address micropollutants, reinforcing the necessity of combining advanced oxidation, adsorption, or membrane-based polishing with biological treatment (Li et al, 2022; Asheghmoalla & Mehrvar, 2024). With respect to microplastics, recent field-scale studies report robust and consistent removal rates (>99%) in advanced plants, largely unaffected by seasonal variability, thereby validating the potential of hybrid systems to ensure stable effluent quality under fluctuating environmental conditions (Iordachescu et al, 2024).

RESEARCH GAP: Despite growing evidence of the technical promise of bio–AOP systems, the literature remains fragmented. Most studies emphasize pollutant removal efficiencies or modeling outcomes, with limited comprehensive evaluations that simultaneously assess contaminant removal, microbial community responses, and sustainability indicators such as energy use, sludge yield, and carbon footprint. This gap is particularly critical in water-stressed regions such as the Middle East and North Africa, where sustainable wastewater reuse is essential for securing water and food resources (Patel & Singh, 2023; Khan et al., 2024).

1 2. Materials and Methods

2.1 Wastewater Source and Characterization

The influent channel of a municipal wastewater treatment facility (Sabha Wastewater Treatment Plant) was used to gather samples of raw wastewater. A typical combination of light industrial discharges and household sewage from a medium-sized urban area made up the influent. Using an automated sampler, composite samples were collected over a 24-hour period to account for diurnal variations in flow and pollutant loading. After being homogenized, the collected samples were kept at 4 °C for a maximum of 48 hours before being used. Microplastics, heavy metals, nutrients, conventional pollutants, and medications were measured as part of the baseline characterization of the influent. The average values (mean \pm SD, range) were as follows: COD 518 \pm 23 mg/L (492–545 mg/L), BOD₅ 242 \pm 18 mg/L (221–265 mg/L), TN 42.1 \pm 2.5 mg/L (39–46 mg/L), TP 7.4 \pm 0.6 mg/L (6.8–8.3 mg/L), Pb 35.2 \pm 2.1 μ g/L (32–38 μ g/L), Cd 4.3 \pm 0.5 μ g/L (3.6–5.0 μ g/L), carbamazepine 2.4 \pm 0.2 μ g/L (2.1–2.6 μ g/L), diclofenac 1.8 \pm 0.1 μ g/L (1.6–1.9 μ g/L), and microplastics 1215 \pm 85 particles/L (1104–1342 particles/L). These values served as reference points for calculating pollutant removal efficiencies.

2.2 Experimental Setup

2.2.1 Biological Treatment Unit

A laboratory-scale continuous-flow activated sludge (CFAS) reactor with a working volume of 20 L was constructed from transparent acrylic material. The system was equipped with:

- Fine-bubble aeration to maintain dissolved oxygen (DO) at 2–3 mg/L.
- A mechanical stirrer to ensure homogeneous mixing.
- A settling zone for solid–liquid separation.
- Temperature control maintained at 22 \pm 2 °C.

The reactor was inoculated with activated sludge sourced from the same WWTP and acclimatized for two weeks prior to experimentation to stabilize microbial communities.

2.2.2 Hybrid Treatment Unit (Bio + AOP)

The hybrid treatment configuration integrated the same biological unit with a downstream advanced oxidation process (AOP) chamber operated in batch mode. The AOP reactor was a 5 L cylindrical photoreactor equipped with:

- A medium-pressure mercury UV lamp system adjustable to 200, 400, or 600 W/m².

- A peristaltic dosing system for hydrogen peroxide (H_2O_2) at concentrations of 10, 45, or 80 mg/L.
- A quartz sleeve to maximize UV light penetration.
- pH control at 6.0, 7.2, or 8.4 using 0.1 M NaOH or H_2SO_4 .
- Adjustable influent flow rates to achieve hydraulic retention times (HRTs) of 4, 8, or 12 h.

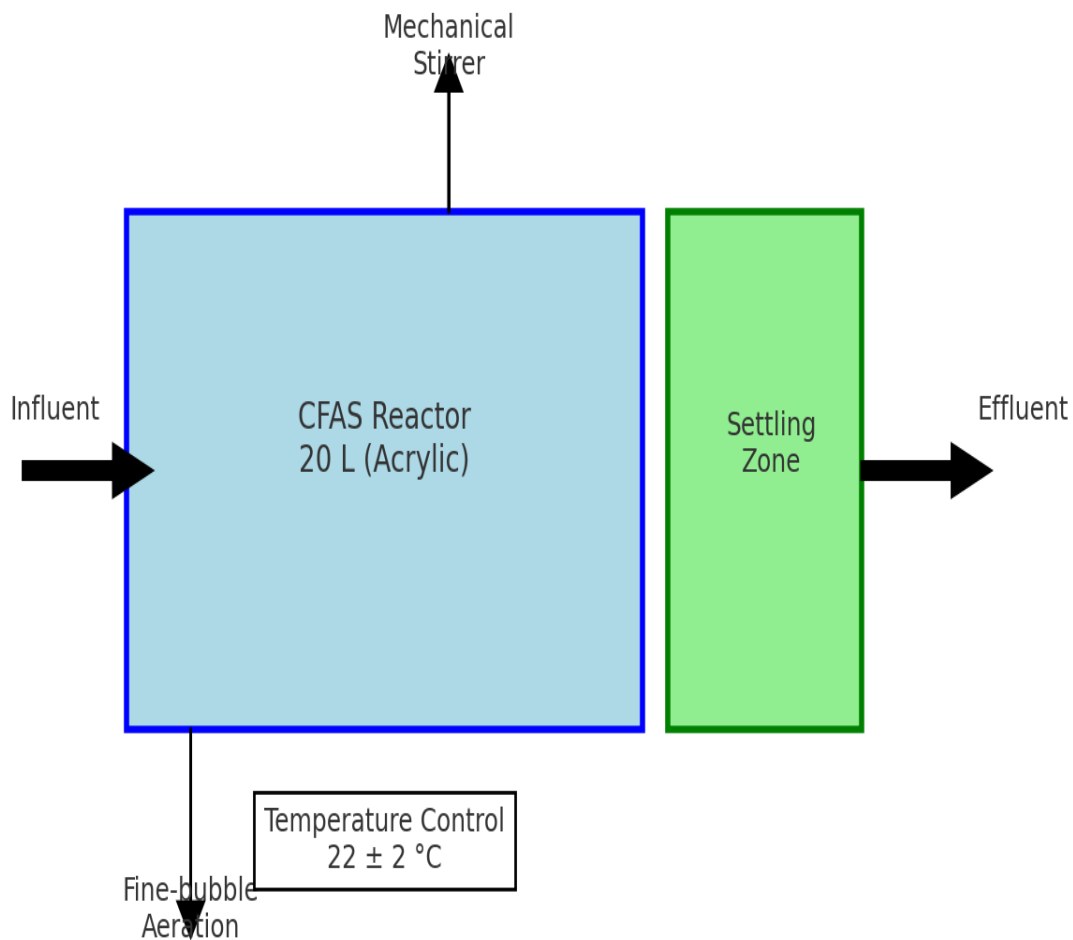


Figure 2 schematic of laboratory – scale CFAS Reactor (20 L)

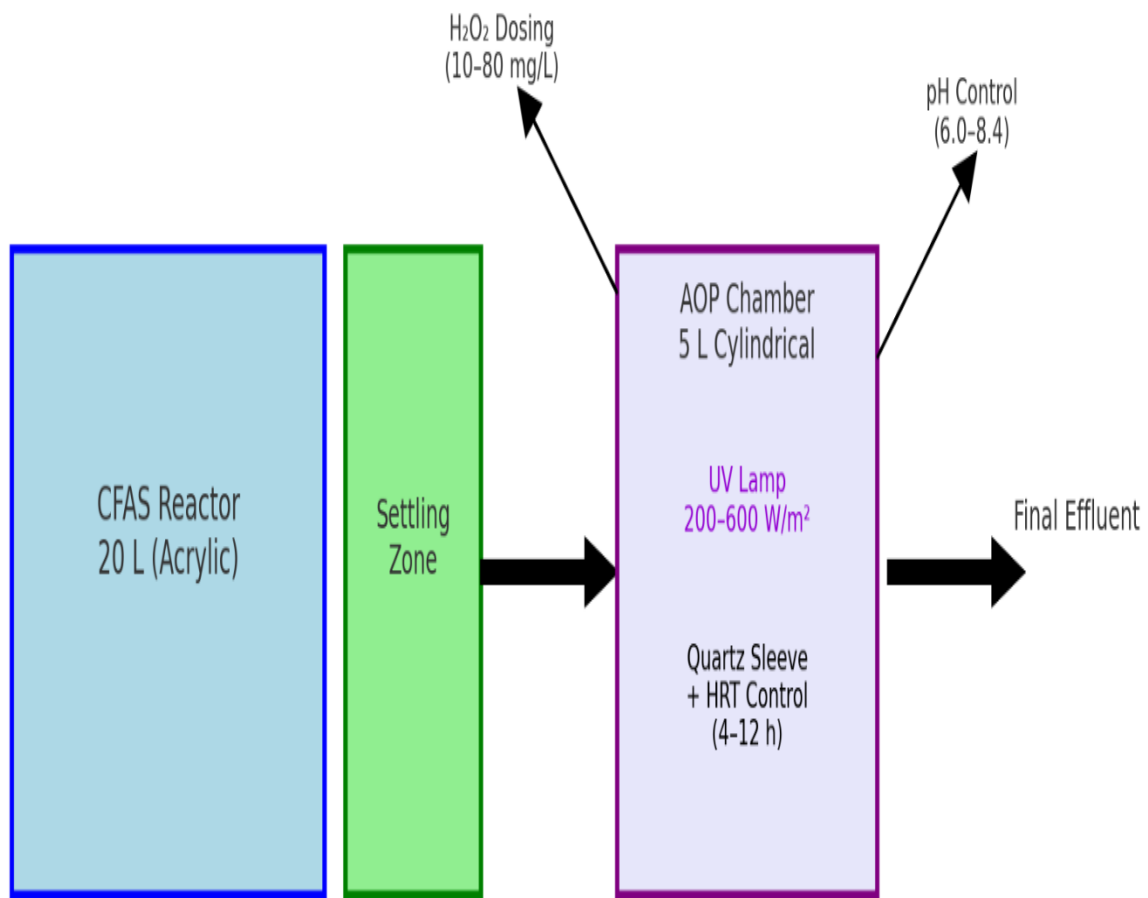


Figure 3 schematic of Hybrid Bio-AOP Treatment System

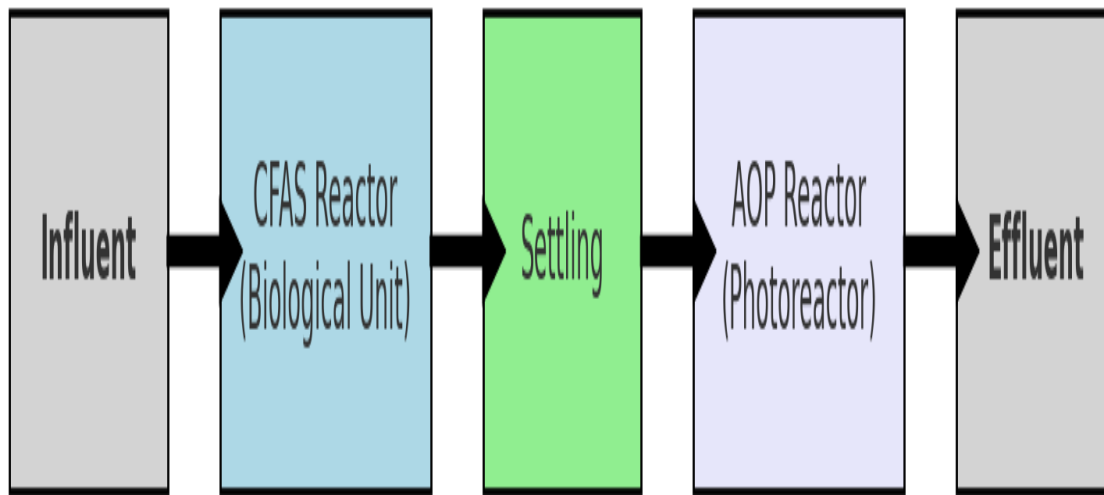


Figure 4 flow diagram of Hybrid Bio-AOP Treatment Process

2.3 Experimental Design and Procedure

A **full factorial experimental design** ($3 \times 3 \times 3 \times 3$) was employed to systematically evaluate the influence of four operational factors: pH, HRT, H_2O_2 dosage, and UV intensity, each at three levels. This resulted in 81 unique treatment conditions.

Two parallel treatment trains were operated:

1. **Biological-only control:** continuous operation of the CFAS unit.
2. **Hybrid bio–AOP system:** effluent from the CFAS unit directed into the AOP chamber.

Each experimental condition was performed in triplicate ($n = 3$) to ensure reproducibility, and results are reported as mean \pm standard deviation.

2.4 Analytical Methods and Quality Assurance/Quality Control (QA/QC)

Analytical procedures followed **APHA (2017)** and **USEPA (Method 200.8)** protocols. Key methods included:

- COD: Closed reflux, colorimetric (APHA 5220D).
- BODs: 5-day incubation at $20\text{ }^\circ\text{C}$ (APHA 5210B).
- TN: Persulfate digestion and UV spectrophotometry (APHA 4500-N).

- TP: Ascorbic acid method (APHA 4500-P).
- Pb and Cd: ICP-MS (Agilent 7700).
- Pharmaceuticals: HPLC with C18 column, 254 nm detection (Agilent 1260).
- Microplastics: Density separation, membrane filtration (0.45 µm), and stereomicroscopy enumeration.

QA/QC measures included:

- Calibration of instruments before each analytical batch.
- Use of blanks and duplicate samples for every parameter.
- Method blanks and spike recoveries for pharmaceutical and microplastic analyses.
- Analytical precision maintained within ±5%.

2.5 Operational and Sustainability Metrics

Operational and environmental performance was assessed based on:

- **Energy consumption (kWh/m³):** calculated from electrical input of aeration pumps and UV lamps relative to treated volume.
- **Operating expenditure (OPEX, USD/m³):** estimated from electricity, chemical consumption (H₂O₂, pH adjusters), and sludge disposal.
- **Sludge yield (kg/m³):** measured by drying sludge samples at 105 °C to constant weight.
- **Global warming potential (GWP)** represents the carbon footprint of the treatment process, expressed in kg CO₂-equivalents per cubic meter. It was estimated using electricity emission factors (IPCC, 2014).

2.6 Data Analysis

Pollutant removal efficiency (%) was calculated as:

$$\text{Removal Efficiency (\%)} = \frac{C_{in} - C_{out}}{C_{in}} \times 100$$

where C_{in} is the influent concentration and C_{out} is the effluent concentration.

Statistical significance was tested using one-way ANOVA followed by Tukey's post-hoc test ($p < 0.05$). Multi-criteria performance evaluation was applied to identify optimal operating conditions by balancing pollutant removal, energy demand, and cost efficiency.

3. Results

3.1. Influent Wastewater Characteristics

The influent wastewater reflected typical municipal and light industrial contributions. Mean \pm SD concentrations were COD 518 ± 23 mg/L, BOD₅ 242 ± 18 mg/L, TN 42.1 ± 2.5 mg/L, TP 7.4 ± 0.6 mg/L, Pb 35.2 ± 2.1 μ g/L, Cd 4.3 ± 0.5 μ g/L, carbamazepine 2.4 ± 0.2 μ g/L, diclofenac 1.8 ± 0.1 μ g/L, and microplastics 1215 ± 85 particles/L. These values established the baseline for pollutant removal calculations.

3.2. Pollutant Removal Performance

3.2.1. Bio-only vs. Hybrid Systems

Removal efficiencies are compiled for both configurations in Table 1. The hybrid system frequently performed better than the bio-only control ($p < 0.05$). COD and BOD₅ removal exceeded 90 percent in the hybrid configuration compared with ~60–70 percent in the bio-only line. Pharmaceuticals and microplastics poorly removed biologically were reduced by $> 70\%$ receiving hybrid therapy.

Table 1. Pollutant removal efficiencies (mean \pm SD, %) in biological-only and hybrid systems

Parameter	Influent (mean)	Bio-only (%)	Hybrid (%)
COD (mg/L)	518	62.5 ± 3.1	89.7 ± 2.8
BOD ₅ (mg/L)	242	71.2 ± 2.6	94.5 ± 2.2
TN (mg/L)	42.1	28.3 ± 1.9	55.7 ± 2.4
TP (mg/L)	7.4	22.1 ± 1.1	48.2 ± 1.9
Pb (μ g/L)	35.2	38.6 ± 2.2	74.9 ± 2.5
Cd (μ g/L)	4.3	32.4 ± 2.0	71.5 ± 2.1
Carbamazepine	2.4	18.7 ± 1.4	72.6 ± 2.6
Diclofenac	1.8	21.4 ± 1.3	70.8 ± 2.2

Parameter	Influent (mean)	Bio-only (%)	Hybrid (%)
Microplastics	1215	25.2 ± 1.7	79.3 ± 3.1

3.2.2. Effect of pH

Performance peaked at near-neutral pH (7.2). COD and BOD₅ removals were 6–10% higher at pH 7.2 than under acidic (6.0) or alkaline (8.4) conditions. Pharmaceutical removal was highly pH sensitive: diclofenac removal reached 77 ± 2.3% at pH 7.2 but dropped to 61 ± 2.0% at pH 8.4.

3.2.3. Effect of Hydraulic Retention Time (HRT)

Longer HRTs enhanced removal. At 4 h, COD and BOD₅ removals were 78 ± 3% and 85 ± 2%, respectively. Increasing HRT to 8–12 h improved removals above 90% for COD/BOD₅ and >70% for pharmaceuticals. Microplastics removal also improved with time. Gains beyond 8 h diminished relative to reactor volume demands.

3.2.4. Effect of Oxidant Dose and UV Intensity

H₂O₂ dosage significantly influenced removal. At 10 mg/L, pharmaceutical removal averaged only 45 ± 2%, while 45 mg/L yielded >70%. At 80 mg/L, no proportional benefits were observed due to hydroxyl radical scavenging. UV intensity of 400–600 W/m² improved breakdown, though benefits plateaued above 400 W/m².

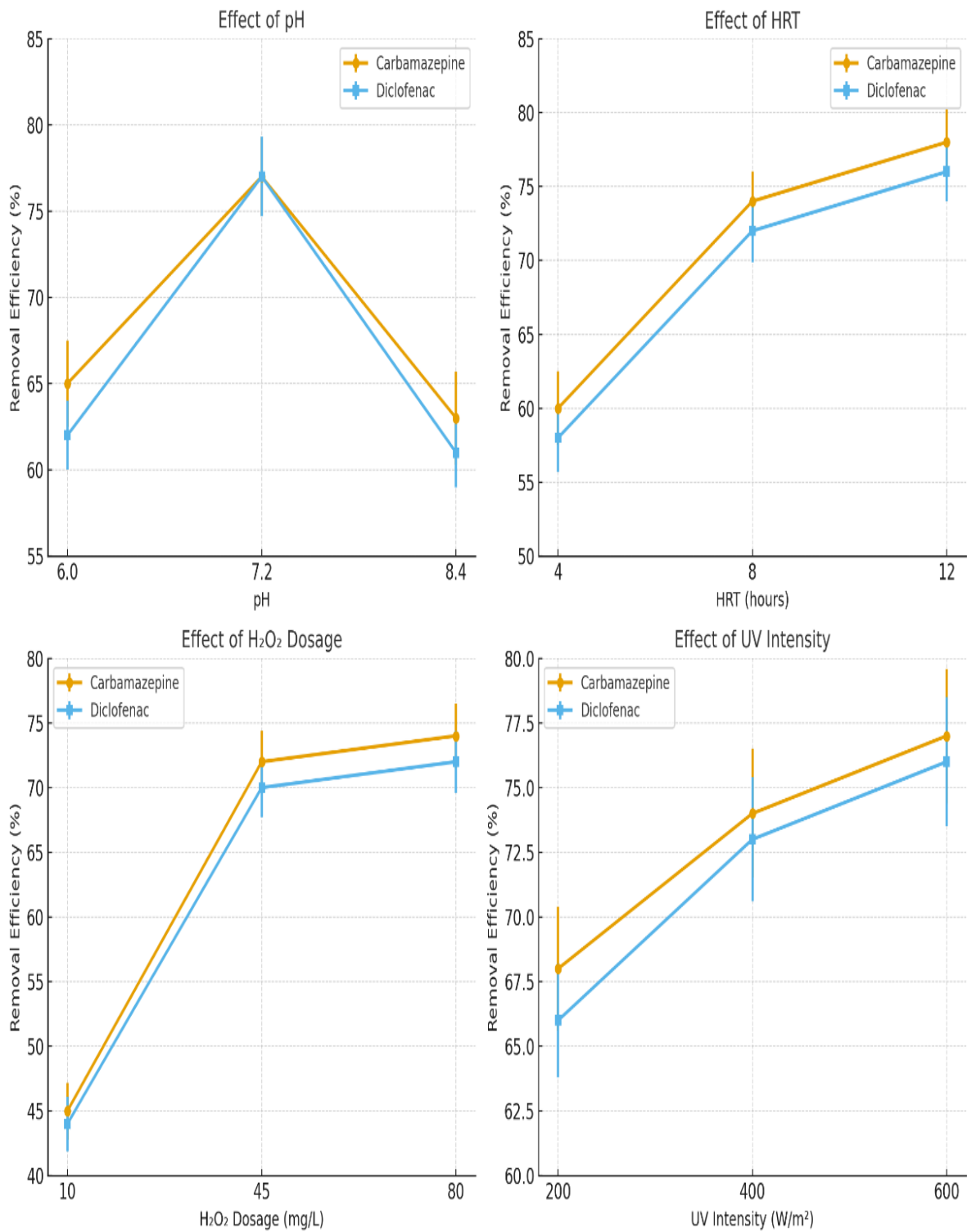


Figure 5 operational factors affecting pharmaceutical removal (Hybrid system)

3.3. Operational and Sustainability Metrics

Operational indicators are compared in Table 2. The hybrid system reduced the carbon footprint by about 34% and produced 30% less sludge, despite using more energy and incurring higher OPEX. Energy consumption per 1 percent COD removal was computed to normalize the results. In comparison to 0.0021 kWh/m³/percent for bio-only treatment, the hybrid system used 0.0026 kWh/m³/percent. The hybrid system's operating expenditure (OPEX), which is the total running cost of treatment per cubic meter, was marginally higher (0.0013 vs. 0.011% USD/m³/percent). These normalized values demonstrate that the hybrid system was only slightly less cost-efficient in comparison to removal performance, even though its absolute costs were higher.

Table 2. Operational and sustainability metrics (mean \pm SD) for bio-only and hybrid systems

Metric	Bio-only	Hybrid	Normalized per % COD removal
Energy (kWh/m ³)	0.13 \pm 0.01	0.29 \pm 0.02	0.0021 vs. 0.0026
OPEX (USD/m ³)	0.07 \pm 0.004	0.12 \pm 0.006	0.0011 vs. 0.0013
Sludge yield (kg/m ³)	0.46 \pm 0.03	0.32 \pm 0.02	–
GWP (kg CO ₂ e/m ³)	0.38 \pm 0.02	0.25 \pm 0.01	–

3.4. Statistical Analysis

For all pollutants, a one-way ANOVA verified that the removal efficiencies of hybrid and bio-only systems differed significantly ($p < 0.05$). While HRT had a significant impact on nutrient removal, Tukey's test revealed that pH and H₂O₂ dose had the greatest influence on pharmaceuticals.

3.5. Optimal Operating Conditions

The optimal balance of efficiency and sustainability was achieved at pH 7.2, HRT 8 h, H₂O₂ dose 45 mg/L, and UV 400 W/m². Under these conditions, the hybrid system achieved COD 91.3 \pm 2.7%, BOD₅ 95.4 \pm 2.3%, TN 59.2 \pm 2.5%, TP 50.1 \pm 1.9%, Pb 76.2 \pm 2.4%, Cd 73.4 \pm 2.1%,

carbamazepine $77.8 \pm 2.6\%$, diclofenac $75.6 \pm 2.3\%$, and microplastics $81.5 \pm 3.0\%$. Energy use was 0.24 kWh/m^3 , with a carbon footprint of $0.23 \text{ kg CO}_2\text{e/m}^3$.

4. Discussion

The hybrid biological–AOP system demonstrated superior performance compared with the biological only baseline, confirming the synergistic value of integrating microbial degradation with oxidative polishing. COD and BOD₅ reductions above 90% reflect the complementary roles of the two stages: the biological unit removed readily degradable organics, while the AOP stage oxidized persistent contaminants, ensuring compliance with stringent effluent standards. Similar enhancements have been reported in recent hybridization studies (Oller et al., 2011; Martins et al., 2022).

4.1 Organic Matter and Nutrient Removal

The nearly twofold improvement in nutrient removal relative to the bio only unit underscores the system's potential for mitigating eutrophication risks. Oxidative pre-treatment likely enhanced nutrient bioavailability and improved microbial assimilation, consistent with earlier findings by Sharma et al. (2020). Notably, these improvements were achieved under moderate operational settings (pH 7.2, H₂O₂ ~45 mg/L), suggesting scalability with optimized design.

4.2 Pharmaceuticals and Microplastics

Drugs that are known to persist in normal biological processes, like diclofenac and carbamazepine (Margot et al., 2015), were eliminated in the hybrid system by more than 70%. The removal of microplastics surpassed 80%, most likely as a result of oxidation, photolytic fragmentation, and aggregation-enhanced settling. These findings collectively show that hybrid systems are capable of efficiently addressing particulate contaminants and dissolved micropollutants, two issues that traditional WWTPs fall short in addressing.

4.3 Heavy Metals

The >70% removal of Pb and Cd suggests oxidative transformation of metal complexes and precipitation within bioflocs (Fu & Wang, 2011). This highlights the applicability of hybrid bio–AOP systems in treating not only municipal wastewater but also industrial effluents with trace metals.

4.4 Comparative Benchmark with Alternative Technologies

Compared to other advanced hybrid strategies, bio–AOP systems exhibit a favorable balance of efficiency and sustainability. For instance:

- **Biological + Membrane filtration:** Achieves high removal (>95% organics, >90% pharmaceuticals) but is limited by membrane fouling, high energy demand (>0.6–1.0 kWh/m³), and brine management costs (Gao et al., 2021).
- **Biological + Adsorption (Activated Carbon):** Provides efficient removal of pharmaceuticals (>85%) at lower energy cost (~0.15 kWh/m³), but requires frequent regeneration, with adsorbent costs ranging from 0.08–0.15 USD/m³ treated (Nguyen et al., 2020).
- **Hybrid Bio–AOP (this study):** Achieved broad-spectrum removal (>70% of ECs, >90% COD/BOD₅) with moderate energy demand (0.24–0.29 kWh/m³) and reduced sludge yield (–30%).

These benchmarks indicate that bio–AOP systems, while slightly costlier than adsorption, offer broader removal capability and lower sludge burden than membranes, making them competitive for sustainable reuse.

4.5 Operational Efficiency and Sustainability

Despite higher energy and chemical inputs, the hybrid system achieved a 34% reduction in carbon footprint compared with biological only treatment. Normalized performance metrics revealed that the additional operational costs were marginal when considered relative to pollutant removal. This supports the hybrid system as a sustainable option where water reuse and environmental compliance are prioritized.

4.6 Practical Applications

The ability of hybrid bio–AOP systems to generate effluents of high quality makes them promising for diverse reuse applications, including agricultural irrigation, industrial processes such as cooling, and even indirect potable supply. These applications are especially valuable in arid and semi-arid areas, notably in the Middle East and North Africa, where water scarcity poses a major challenge.

4.7 Limitations and Future Research Directions

This study highlights important advantages but also several limitations:

1. **Scale-up challenges:** Laboratory findings may not fully translate to pilot- or full-scale systems due to higher energy demands of UV/H₂O₂ photoreactors. Future work should explore energy-efficient alternatives, such as UV-LEDs or solar-driven AOPs.
2. **By-product formation:** Residual oxidants and transformation by-products (e.g., aldehydes, bromate) were not monitored in this study. Advanced analytical tools (e.g., HRMS) should be employed to assess potential health risks of secondary pollutants.
3. **Integration into WWTPs:** Retrofitting hybrid systems requires evaluation of hydraulic compatibility, spatial constraints, and techno-economic feasibility compared with alternative tertiary treatments.
4. **Long-term resilience:** Further research should investigate system stability under variable influent loads, seasonal changes, and shock conditions, including microbial community dynamics over time.
5. **Health and risk assessment:** Beyond pollutant removal, future research should include **ecotoxicity tests** and quantitative risk assessments to evaluate the safety of effluents intended for reuse.

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