




Advanced oxidative processes in the degradation of emerging pollutants: Drugs and dyes in aqueous solutions

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ABSTRACT

The increase in industrial, pharmaceutical, and food consumption has intensified the presence of emerging contaminants (ECs) in water bodies, including antibiotics such as oxytetracycline (OTC) and synthetic dyes such as Blue 19, known for their high persistence and toxicity in the environment. This problem represents an environmental and health challenge due to the difficulty of degrading these substances using conventional methods. The objective of this research was to evaluate the effectiveness of homogeneous advanced oxidation processes (AOP) in the degradation of OTC and Blue 19 in aqueous solutions. To this end, a completely randomized design with five treatments (UV/O₃, UV/H₂O₂, UV/O₃/H₂O₂, photo-Fenton, and O₃) was applied, also considering kinetic modeling using the Chan and Chu model and toxicity bioassays with carrot seeds (*Daucus carota*). The results showed that ozone (O₃) achieved the highest efficiency, with 100% removal of Blue 19 in 20 minutes and an 89.97% reduction in OTC in 120 minutes. However, the bioassays revealed that the formation of by-products had inhibitory effects on germination, highlighting the need to evaluate not only the efficiency of AOPs but also the residual toxicity of the treatments. In conclusion, AOPs, and ozone in particular, represent an effective alternative for reducing emerging pollutants, although their application parameters need to be optimized and integrative processes considered to minimize the impacts of the by-products generated.

Keywords: emerging contaminants, oxytetracycline, advanced oxidation processes, toxicity, ozone, dyes.

INTRODUCTION

The presence of emerging contaminants (ECs) in water bodies has attracted growing scientific and social interest due to their persistence, bioaccumulation, and adverse effects on human health and aquatic ecosystems (Vasilachi et al., 2021; Senthil et al., 2024; Singh et al., 2024). These compounds, which include antibiotics and synthetic dyes, have increased in parallel with the rise in industrial, pharmaceutical, and food consumption (Arbelaez, 2015; Akhil et al., 2021; Duré et al., 2022; Boyko et al., 2023; Osuna and Moreno, 2024; Li et al., 2025). It is estimated that in developing countries, up to 90% of wastewater is discharged without prior treatment, which intensifies the dispersion of ECs in rivers, wetlands,

and vulnerable ecosystems (Arias et al., 2023; Tella et al., 2025).

Several studies have pointed out that, of the more than 100 million chemicals identified in water globally, at least half are emerging pollutants, including pharmaceuticals and dyes (Gayosso & González, 2021). More than 150 types of pharmaceuticals have been reported in aquatic environments, even in remote regions such as the Arctic (Kallenborn et al., 2018 Juárez et al., 2023). In turn, the textile industry produces more than 700,000 tons of dyes with more than 10,000 chemical varieties each year, which increases the challenge of treating them in wastewater (Zaruma et al., 2018).

Among antibiotics, oxytetracycline (OTC) is one of the most widely used drugs in aquaculture due to its effectiveness against bacterial, fungal,

and viral infections. It belongs to the tetracycline group and its mechanism of action consists of inhibiting protein synthesis by binding to the 30S ribosomal subunit (Huang et al., 2025). OTC has been detected in concentrations of up to 1200 µg/L in hospital effluents and 85 mg/kg in soils amended with manure (Huang et al., 2025).

In Latin America, recent studies document its intensive use in the shrimp industry in Ecuador, Mexico, and Brazil, which increases environmental pressure on coastal ecosystems and wetlands (Serna et al., 2018; Sotelo et al., 2021; Corbalá et al., 2024). The case of the La Segua Wetland in Manabí, Ecuador, is a representative example of the bioaccumulation of antibiotics in sediments and groundwater due to aquaculture activity (Rodríguez et al., 2020; Chancay et al., 2021). In addition to its direct toxic effects, OTC contributes to the spread of antimicrobial resistance genes, which has been identified by the WHO as a critical threat to public health.

At the same time, synthetic dyes such as Reactive Blue 19 (AR19 or RB19) are another category of CE of high concern. Widely used in the textile, food, and pharmaceutical industries, they have recalcitrant azo structures, high solubility, and resistance to conventional chemical and biological processes (Domingues et al., 2024; Periyasamy, 2024). Their environmental persistence has been documented for more than four decades, as has their ability to generate mutagenic and carcinogenic by-products (Banaei et al., 2017; Ismail et al., 2019; Almeida et al., 2024; Islam et al., 2025).

In countries such as Ecuador, textile effluents reach daily volumes of more than 14,000 liters with a high pigment load, which exacerbates water pollution (Ramos et al., 2018; Núñez et al., 2023). The coexistence of antibiotics and dyes in aquatic matrices creates a synergistic environmental risk, as both can interact to produce secondary metabolites with toxicity greater than that of the original compounds (Ji et al., 2025; Adnan et al., 2025).

These synergistic effects impact microbial diversity, alter biogeochemical cycles, and threaten ecological stability (Battauz, 2021; Ogidi, 2022; Pérez et al., 2024). In addition, sublethal impacts on aquatic organisms have been reported, such as endocrine disruption, decreased fertility, and immune dysfunction (Samuelsen et al., 2021; Vergara et al., 2024).

The situation is even more critical in regions such as Latin America, where rapid growth in aquaculture and the textile industry has intensified

CE discharges. In Ecuador, shrimp production accounts for more than 40% of national exports and has been accompanied by the extensive use of antibiotics since the 1970s (Piedrahita, 2018; Gonzabay et al., 2021). At the same time, the textile industry is concentrated in provinces such as Pichincha, Guayas, and Tungurahua, where industrial effluents generate a growing load of dyes and pigments (Estupiñán, 2020; Mendoza et al., 2022).

Given this situation, advanced oxidation processes (AOPs) have emerged as technological alternatives with great potential for the remediation of CE-contaminated water. These processes are based on the generation of hydroxyl radicals and reactive oxygen species, capable of mineralizing recalcitrant compounds that conventional methods cannot eliminate (Nidheesh et al., 2022). Among the most studied variants are Fenton, photo-Fenton, UV/H₂O₂, ozonation, and heterogeneous photocatalytic processes (Manrique et al., 2017; Akbar et al., 2023; Almeida et al., 2024; Ramírez et al., 2025). Their combination with traditional methods, such as coagulation-flocculation, enhances the removal of organic matter and pathogenic microorganisms (López et al., 2021; Naranjo et al., 2021). In addition, recent advances highlight their efficiency, relative low cost, and scalability, which facilitates their implementation even in developing regions (Polo & Sánchez, 2021; Ramírez et al., 2025; Hadi et al., 2021).

The importance of POAs also lies in their alignment with the Sustainable Development Goals (SDGs). They contribute to SDG 6 by improving water quality, to SDG 12 by promoting the use of sustainable technologies, and to SDG 14 by reducing toxic risks to marine biodiversity. They also support SDG 3, related to health, and SDG 9, aimed at innovation in treatment systems (United Nations, 2015).

In this context, this research seeks to evaluate the application of AOP for the degradation of EC in aqueous solutions, thus contributing to the development of sustainable technological solutions to the challenges of water pollution.

MATERIALS AND METHODS

Chemicals

The commercial grade Blue 19 dye was purchased from Aromcolor Ecuador. Oxytetracycline hydrochloride (HPLC grade ≥95%)

was obtained from Sigma-Aldrich (Germany). 30% hydrogen peroxide (v/v) was provided by Fisher Scientific (United States) and iron (II) sulfate heptahydrate (98.5% purity) by Loba Chemie (India).

Bench reactor

For the study, a bench-top reactor with ultraviolet radiation (UV-C: $\lambda = 100\text{--}280\text{ nm}$) and sunlight was used. The reactor was designed as described by Gorozabel (2022). and was equipped with three 30 W UV-C lamps (Philips) installed in parallel, providing a light intensity of $1.98 \times 10^{-3}\text{ W cm}^{-2}$.

Identification and quantification of the contaminant

Prior to identification and quantification, a binary aqueous solution composed of oxytetracycline hydrochloride ($\geq 95\%$) and Blue 19 dye was prepared, with concentrations ranging from $20\text{ mg}\cdot\text{L}^{-1}$ to $140\text{ mg}\cdot\text{L}^{-1}$, diluted in ultrapure water. Before applying the treatments, the initial concentration of the contaminants in this solution was measured and the corresponding calibration curve was prepared (López and Zambrano, 2024). To determine the concentrations reached by both compounds, a Thermo Scientific 60S Evolution UV/VIS spectrophotometer was used to identify the wavelength and peak absorbance (λ_{max}) of each contaminant. In accordance with previous studies, it was confirmed that oxytetracycline had a λ_{max} of 354 nm and Blue 19 had a λ_{max} of 594 nm (López and Zambrano, 2024).

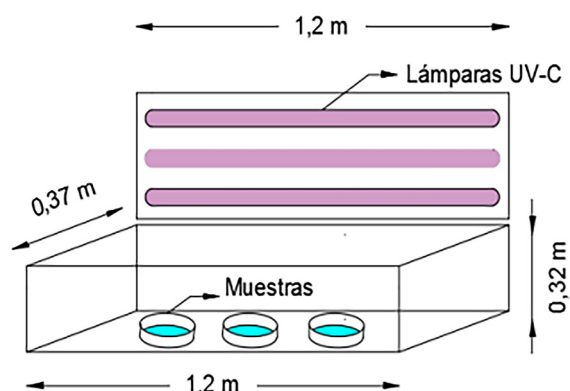


Figure 1. UV-C bench reactor
Source: Gorozabel (2022)

Experimental design

Five treatments (UV/O₃, UV/H₂O₂, UV/O₃/H₂O₂, Photo-Fenton, and O₃) were evaluated, with three replicates of each treatment, under a completely randomized design (CRD) with a single factor, which allowed for control of the influence of external variables. Each experimental unit consisted of an Erlenmeyer flask with 500 mL of the binary solution contaminated at $100\text{ mg}\cdot\text{L}^{-1}$, treated in a bench reactor according to the corresponding POA (Gorozabel et al., 2021; Pinoargote et al., 2022; Giler et al., 2020). However, the assumptions of normality and homoscedasticity were verified before applying an ANOVA, followed by Tukey's test ($p < 0.05$), to identify significant differences between treatments and determine the most effective in degrading the dye and the drug.

Kinetic modeling

Solutions with varying concentrations of 20, 60, 100, 140, 180, 220, and 260 $\text{mg}\cdot\text{L}^{-1}$ were used. These were placed in Erlenmeyer flasks and stirred at 200 rpm, maintaining a constant temperature of 298 K (25°C). Measurements were taken at intervals of 5, 10, 15, 20, 30, 40, 50, 60, 90, and 120 minutes, depending on the treatment applied, using UV spectrophotometry (Gorozabel, 2022). For process modeling, the methodology proposed by Chan and Chu (2003) and Giler et al. (2020) was followed, applying a pseudo-first-order kinetic model to describe degradation.

$$C = C_0 \cdot \left(1 - \frac{t}{\rho + \sigma t}\right) \quad (1)$$

Where C was the final concentration of the oxytetracycline solution after applying the advanced oxidation process at a given time (t), C_0 was the initial concentration of the drug solution ($\text{mg}\cdot\text{L}^{-1}$), and the parameters $1/\rho$ and $1/\sigma$ represented the degradation rate constant (min^{-1}) and

Table 1. Composition of treatments

Treatments	AOPs	Binary solution Oxytetracycline and blue dye 19 (ml)
T ₁	UV/O ₃	500
T ₂	UV/H ₂ O ₂	500
T ₃	UV/O ₃ /H ₂ O ₂	500
T ₄	Photo-Fenton	500
T ₅	O ₃	500

the oxidative capacity of the treatment, respectively, while the values of these parameters were found by linearizing Equation 1, according to Equation 2 (Giler, 2018).

$$\frac{t}{\left(1-\frac{c}{c_0}\right)} = \rho + \sigma t \quad (2)$$

The creation of the graph $\frac{t}{\left(1-\frac{c}{c_0}\right)}$ versus time, allowed us to obtain the respective angular and linear coefficients of the parameters σ and ρ .

Toxicity study

The toxicity of the binary solution (oxytetracycline + Blue 19) was evaluated using germination bioassays with carrot seeds (*Daucus carota*) (Gorozabel, 2022; Reina et al., 2020). Eighty seeds were distributed in eight glass Petri dishes (10 cm) lined with filter paper (12.5 cm) and exposed for 120 h to solutions at 1%, 5%, 10%, 50%, 70%, and 100%, plus water (negative) and 3% boric acid (positive) (Pinoargote et al., 2020; Giler, 2018). Following the recommendations of Gómez (2024), 2 mL of each solution was added per dish and the tests were performed in triplicate, using USEPA protocols for seed germination and root elongation tests (United States Environmental Protection Agency [USEPA], 1996).

After incubation, root growth was evaluated by calculating the relative growth rate (RGR) and

germination index (GI) using Equations 3 and 4, respectively:

$$RGR = \frac{RLS}{RLC} \quad (3)$$

$$GI = RGR \cdot \frac{SGS}{SGC} \cdot 100 \quad (4)$$

Where RLS was the total root length in the sample, RLC was the total root length in the negative control, SGS was the number of seeds germinated in the sample, and SGC was the number of seeds germinated in the negative control.

RESULTS

Identification and quantification of the contaminant

The OTC spectrum showed absorbance maxima at 276 and 354 nm, the latter being selected due to its lower interference with aromatic absorptions and its proximity to the UV-Vis region, in accordance with previous studies. Likewise, Blue 19 presented a characteristic peak at 594 nm, confirmed by spectral scanning analysis. The quantification of both compounds was performed using calibration curves ($R^2 > 0.99$) constructed with standards of known concentration.

In this context, the calibration curve corresponding to OTC showed a linear relationship, expressed by the equation $y = 15334.06x - 194146$,

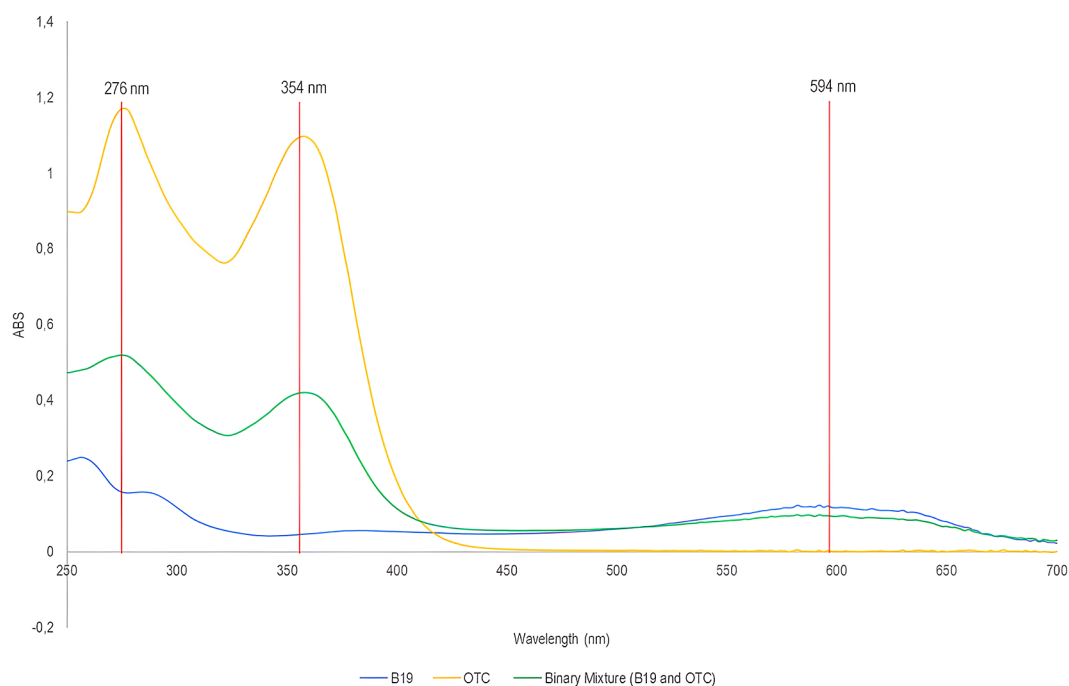


Figure 2. Spectra of emerging contaminants (B19 and OTC) in binary mixture

with a correlation coefficient of $R^2=0.9991$, indicating high accuracy in the evaluated range. Similarly, for the Blue 19 dye, the equation $y=0.0155x+0.1734$ was determined, resulting in a coefficient of determination of $R^2=0.9984$. These values reflect the excellent linearity of the method, as noted by Giler et al. (2020), and confirm the reliability of the concentrations obtained in the samples analyzed.

UV-Vis spectrophotometry proved to be an accurate and reliable tool for the identification and quantification of OTC and Blue 19 in aqueous solutions. Similarly, the choice of 354 nm for OTC minimized interference from aromatic absorptions, supporting the reliability of the method in line with that reported by Sotelo et al. (2021). Azul 19 presented a maximum at 594 nm, consistent with the characteristic absorption of highly conjugated compounds with active chromophores, as described by Martínez and Martínez (2025). The calibration curves showed excellent linearity ($R^2 > 0.99$), which guarantees the reliability of the quantification, while the monitoring of the chromophore bands allows the detection of changes associated with degradation processes, as pointed out by Napoleão et al. (2022). Taken together, these findings confirm that the method applied combines sensitivity, accuracy, and speed, making it an appropriate strategy for monitoring and evaluating emerging contaminants in aquatic systems.

Application of advanced homogeneous oxidative processes

The comparison of the five treatments applied (UV/O₃, UV/H₂O₂, UV/O₃/H₂O₂, Photo-Fenton, and O₃) was carried out using a completely randomized experimental design (CRD), which showed that treatment 5, involving ozone (O₃), achieved the highest efficiency in degrading the pollutants under study. Specifically, 100% elimination of the Blue 19 dye and 89.97% degradation of the OTC concentration were obtained, indicating that this behavior highlights the effectiveness of ozone in removing this type of compound.

This behavior is consistent with previous research, in which ozone has shown high efficiency in removing color from industrial effluents (95–97%), although with limitations in reducing COD and TOC (<50%) (López et al., 2021). In this sense, its high potential for the degradation of emerging pollutants is confirmed, although with limitations in the total mineralization of the solution (Becerra et al., 2021).

With regard to OTC, Giler et al. (2020) indicate that it achieved 97.17% degradation, a value very close to that achieved in the current research with ozone, but they achieved this through the UV/H₂O₂ process. This finding supports the idea that, as in the case of glyphosate, the efficiency of advanced oxidative processes depends on factors such as pH and

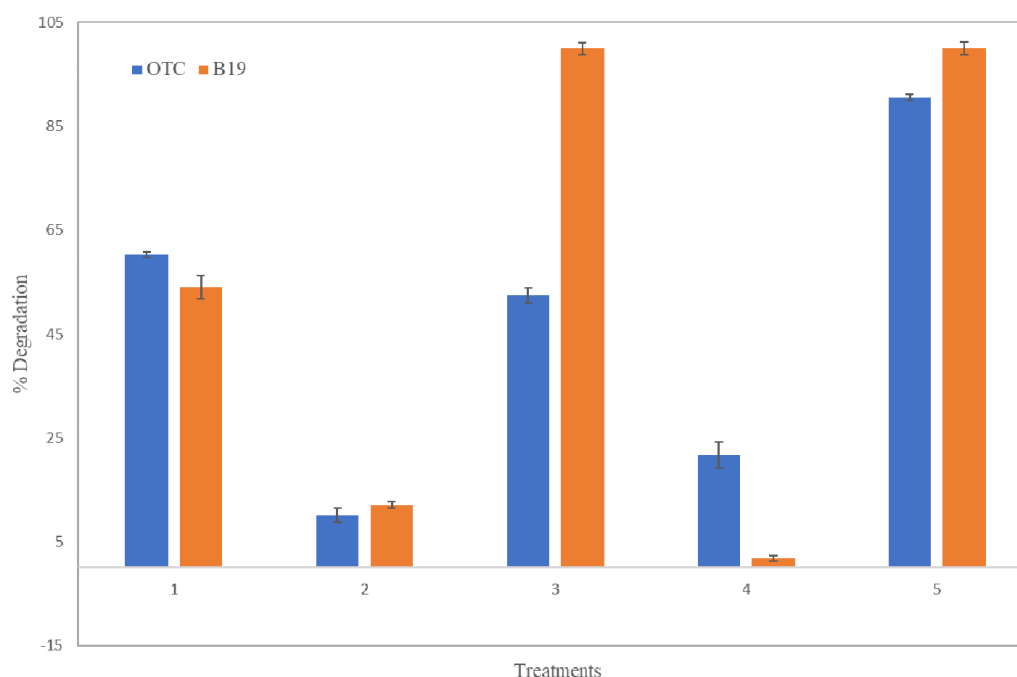


Figure 3. Degradation efficiency of the treatments applied to the binary solution (OTC and B19)

the oxidant present, since under neutral and alkaline conditions, elimination rates of over 96% have been recorded, while in an acidic environment, efficiency is reduced to 60% (de la Cruz, 2025). Taken together, these results reinforce the idea of Pérez & Torres (2024) that ozone is a promising alternative for the removal of dyes and antibiotics in aqueous solutions, although its application must be optimized according to the characteristics of the contaminant and the reaction medium.

Although AOPs are very effective at degrading recalcitrant compounds, their economic viability must be carefully evaluated for application on a wastewater treatment plant (WWTP) scale. Ozonation and UV/H₂O₂ typically incur higher operating costs due to energy consumption (UV lamps) and the continuous input of oxidants (O₃, H₂O₂, or Fe²⁺ salts). Estimates indicate that AOP treatment costs can range from \$0.5 to \$1.5 per m³ of treated water, which is higher than conventional methods such as coagulation-flocculation or activated sludge, whose average costs are \$0.05–0.2 per m³ (Pérez and Torres, 2024; Christou et al., 2024). However, conventional treatments are often ineffective at removing pharmaceuticals and dyes, leaving toxic residues in effluents.

Kinetic modeling

Ozone treatment applied to the binary aqueous solution (OTC and Blue 19) achieved 100% degradation of the Blue 19 dye 20 minutes after application and 89.97% degradation of OTC after 120 minutes, as predicted by the treatment. In terms of the parameters of the applied model shown in Table 2, they reflected values of $1/\rho$ of 0.3164 min⁻¹ for OTC and 0.4388 min⁻¹ for Blue 19. Regarding $1/\sigma$, values of 0.9127 and 0.8454 were reported for OTC and B19, respectively. Likewise, the model fit showed a higher coefficient of determination for OTC ($R^2 = 0.9989$) compared to Blue 19 ($R^2 = 0.9649$) (Figure 3).

The interpretation of these findings suggests that the removal of the dye is attributed to the high susceptibility of aromatic compounds with chromophore groups to the oxidizing action of ozone, which facilitates their structural breakdown. In contrast, OTC showed lower removal efficiency due to the presence of more stable heterocyclic structures, which require greater oxidative demand for their degradation. On the other hand, the better fit of the model to the kinetics of OTC suggests that this contaminant more closely follows the trend described by Chan and Chu (2003), while Blue 19, although completely degraded, shows greater variability in its fit.

Similar results have been reported for Acid Blue 80, where rapid initial kinetics are linked to

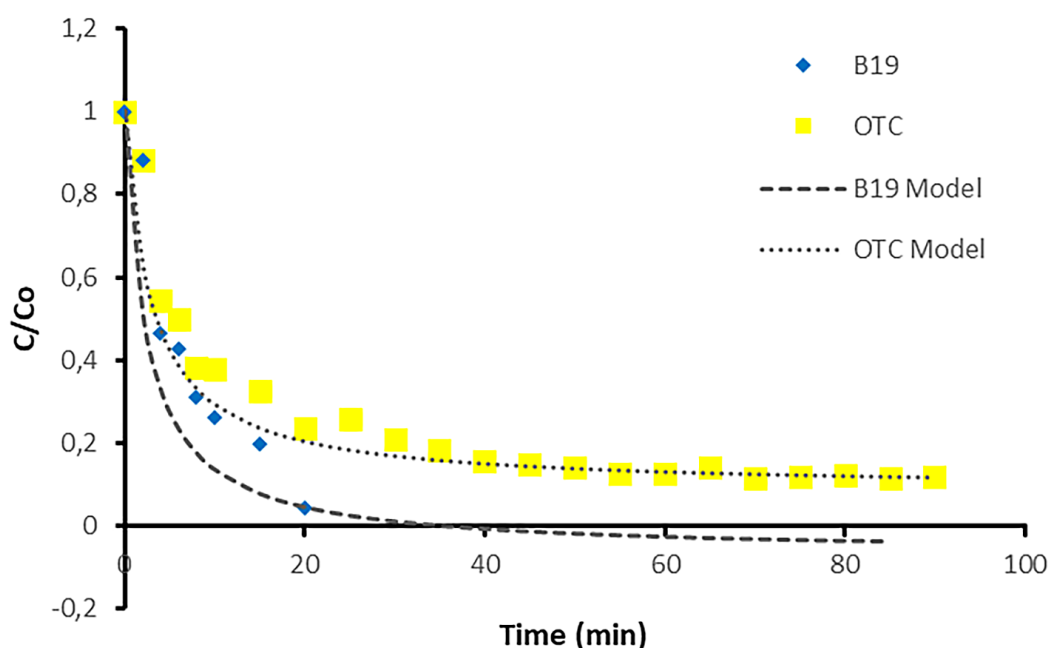


Figure 4. Adjustment of the model proposed by Chan and Chu

Table 2. Parameters of the kinetic model by Chan and Chu (2003) for the treatments used in the degradation of OTC and dye B19 in a binary mixture

Treatment		Degradation (%)	1/p (min ⁻¹)	1/σ	R ²
Ozone (O ₃)	OTC	88.27	0.3164	0.9127	0.9989
	Dye	100	0.4388	0.8454	0.9649

the availability of active sites and linear regression coefficients (R²) greater than 0.9 (Napoleão et al., 2022), confirming the effectiveness of ozone on aromatic compounds and chromophores.

Likewise, in the research conducted by Sotelo et al. (2021), OTC showed an excellent fit to the Chan and Chu model (R² = 0.9989), supported by the linearity of the method with r² = 0.996 in calibration and r² = 0.999 for the complete system, detection and quantification limits of 0.06 and 0.1 µg·mL⁻¹, and a recovery of 104 ± 25.5 % in water and sediment solutions, data that guarantee the reliability of the kinetics obtained, comparable to studies with drugs such as atenolol, which reached 98.73 % degradation after 150 minutes (Andrade et al., 2023), demonstrating that, although contaminants have different degradation rates, kinetic modeling allows their behavior to be predicted and times and doses in ozone treatments to be optimized.

Toxicity study

After applying the ozone treatment, which proved to be the most effective, the toxicity of the substances generated in carrot seeds (*Daucus carota*) during the process was evaluated. To this end, germination tests were carried out using the treatment and, as a control, seeds treated only

with water. The results showed that, in *Daucus carota*, the increase in the concentration of the treatment solution favored the formation of compounds with an inhibitory effect on the development of the species, which was reflected in a reduction in both the number of germinated seeds and root growth (Table 3).

The germination of *Daucus carota* seeds was most affected, suggesting that this species is less resistant to ozone treatment. These findings confirm that, although advanced oxidative treatments generate compounds capable of degrading pollutants, ozone can negatively interfere with the germination and root development of this species. The results obtained are shown in Figures 5, which detail the relative growth rate (RGR) and germination index (GI).

A reduction in root growth of *Daucus carota* was observed in all treatments evaluated (Figure 5). This decrease was more evident as the concentration of the solutions used increased. It should be noted that, under real conditions, effluents discharged into water bodies undergo a dilution process, so their behavior differs from that observed in aqueous solutions with concentrations greater than 50% TSS. The results obtained show that the application of ozone affects both root growth and the germination rate of *Daucus carota*.

Table 3. Mean length of roots, relative growth rate (RGR) and germination index (GI) of carrot (*Daucus carota*) seed according to the concentration of the aqueous OTC and Blue 19 solution used for advanced oxidative treatments

AOP treatment	Ozone (O ₃)			
Species	<i>Daucus carota</i> (carrot)			
Aqueous solution	RG±DP	*RGR	*SS	GI (%)
Water	2.430±0.320	1	9	90
SPT 1%	1.120±0.150	0.461	6	30.73
SPT 5%	0.220±0.150	0.091	6	6.04
SPT 10%	0.130±0.050	0.053	4	2.38
SPT 50%	0.105±0.130	0.043	2	0.96
SPT 70%	0.075±0.110	0.031	1	0.34
SPT 100%	0.050±0.070	0.021	1	0.23
SBT	1.740±0.370	0.716	8	63.65

Note: * SBT – solution before the treatment; SPT – solution post-treatment; SS – sprouted seeds; RG – root growth in cm; RGR – relative growth rate; GI – germination index.

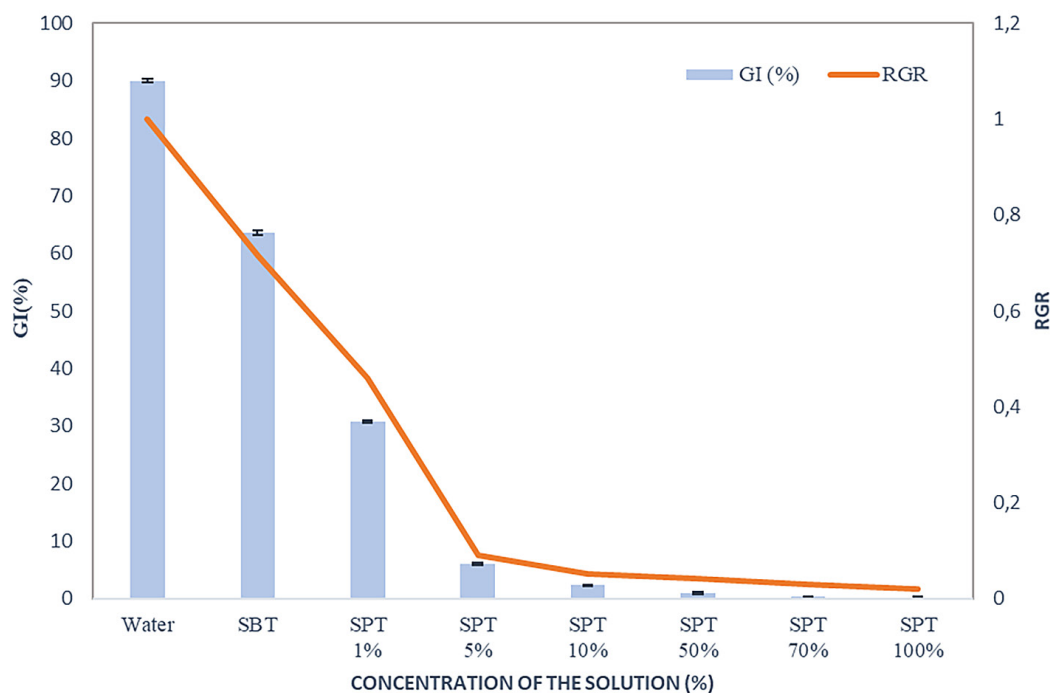


Figure 5. Relative growth rate (RGR) and germination index (GI) of carrot seed (*Daucus carota*) for the treatment with ozone

These results are consistent with previous studies reporting that initial solutions of dyes such as Acid Blue 80 with AOP application show residual toxicity, reflected in lower germination index values compared to the negative control (Napoleão et al., 2022), confirming that the by-products generated during oxidation maintain biological activity and that AOPs, although they eliminate recalcitrant contaminants, must be evaluated considering the effects of intermediates (López et al., 2021).

Likewise, the findings indicate that the by-products formed during ozone treatment significantly affect plant growth, showing marked decreases in the germination index and root growth rate at higher concentrations (Crespo, 2024). This observation is consistent with studies where AOP processes alter contaminant molecules but fail to completely reduce toxicity, suggesting the need for complementary testing in other sensitive organisms such as mollusks and microcrustaceans to assess potential environmental effects (Chiara et al., 2025). Taken together, these results coincide with Andrade et al. (2023) that, although ozone is effective in degrading emerging pollutants, it is essential to monitor the toxicity of the by-products generated to ensure that AOPs do not compromise the health of aquatic and plant organisms.

CONCLUSIONS

The results achieved confirmed that Advanced Oxidation Processes (AOPs) are a highly efficient tool for removing emerging contaminants. Among the treatments evaluated, ozone (O_3) stood out for its speed and effectiveness, achieving total elimination of the dye Blue 19 in just 20 minutes and an 89.97% reduction in oxytetracycline after 120 minutes of exposure. The kinetic fit to the Chan and Chu model supported the reliability of the process, although phytotoxicity bioassays with *Daucus carota* showed the formation of byproducts with negative effects, underscoring the need to optimize operating parameters and consider integrative processes that ensure not only the degradation of primary contaminants but also the neutralization of their derived metabolites.

From a comparative perspective, ozone-based AOPs demonstrated superior performance compared to other homogeneous processes such as UV/ H_2O_2 and photo-Fenton. However, the feasibility of their implementation at the wastewater treatment plant scale requires balancing technical efficiency with economic feasibility, considering both high energy costs and the continuous supply of reagents. In this regard, although POAs far exceed conventional methods (coagulation, activated sludge, filtration) in the removal of persistent

drugs and dyes, they require an optimized design to minimize operating costs and reduce the formation of toxic intermediates.

From a sustainability perspective, AOPs offer significant advantages: (i) broad-spectrum efficacy against pharmaceuticals, dyes, and pathogens, (ii) relatively simple integration into existing treatment infrastructures, and (iii) clear alignment with the Sustainable Development Goals (SDGs), especially SDGs 3, 6, 9, 12, and 14. However, their limitations include high energy consumption, the need for constant chemical inputs, and the generation of potentially harmful by-products. Therefore, future research should focus on the implementation of hybrid systems (AOPs coupled with conventional biological or physicochemical treatments) capable of improving economic profitability and reducing residual toxicity, thus ensuring comprehensive and sustainable management of emerging pollutants in aquatic environments.

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