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Electron beam application to fluoxetine and surfactant mixture degradation, with persulfate, and toxicity approach

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ABSTRACT

Electron beam irradiation (EBI) is an essential tool for removing organic pollutants from water and wastewater. Depending on the type of effluent being treated, this process can be combined with persulfate to enhance removal efficiency. The simultaneous effects of persulfate (PS) initial concentration and absorbed dose on the degradation of the antidepressant fluoxetine were investigated: a binary mixture (fluoxetine and surfactant sodium dodecyl benzene sulfonate) was treated, following a Doehlert experimental design. Toxicity tests were also conducted using *Daphnia similis*. Additionally, the surfactant aqueous solution was treated by an electron beam accelerator. The results showed that 90.1 ± 1.1 % of fluoxetine in the mixture was removed at a dose of 5.0 kGy. The combination of irradiation with persulfate (PS) significantly improved fluoxetine removal compared to EBI, consistently achieving over 90 % removal. However, higher concentrations of persulfate (2.5 mmol L^{-1}) did not increase fluoxetine removal. Toxicity tests were performed on samples treated under the optimized conditions ($[PS]_0 = 1.1$ mmol L^{-1}; absorbed dose - 4.5 kGy) and revealed an increase in *Daphnia similis* toxicity, suggesting the formation of toxic byproducts. Concerning the data obtained for the aqueous solution of irradiated surfactant, more than 90 % degradation was achieved in the evaluated doses (2.5 and 5.0 kGy), resulting in a 70 % reduction in toxicity at 5.0 kGy.

1. Introduction

Pharmaceuticals are crucial for modern healthcare, designed to prevent, cure, and improve the quality of life for humans and animals. Nevertheless, the increased use and disposal of these products have led to concerns about their potential impact on public health and ecosystems, resulting in adverse effects on organisms and the disruption of natural food chains (Hernández-Tenorio et al., 2022; Anand et al., 2022).

Various sources of pharmaceuticals in the environment include improper disposal practices, human and animal excretion, effluents from wastewater treatment plants, waste and residues from hospitals and healthcare facilities, and primarily from pharmaceutical industries (Karungamye et al., 2022). Pharmaceutical manufacturing facilities can be a significant source of pharmaceutical contamination due to the high concentrations of pharmaceuticals in their effluents (Patel et al., 2019). High concentrations of ibuprofen (0.70–1.67 mg L^{-1}), levofloxacin (0–6.20 mg L^{-1}), ciprofloxacin (3.0–5.25 mg L^{-1}), ofloxacin (2.45–4.12 mg L^{-1}), oxytetracycline (0–9.40 mg L^{-1}), and doxycycline (1.58–6.75 mg L^{-1}) have been reported in wastewater from pharmaceutical manufacturing units (Ashfaq et al., 2017; Hussain et al., 2016).

The COVID-19 pandemic has altered lifestyles and impacted psychological disorders such as post-traumatic stress disorder, insomnia, and anxiety among healthcare professionals and the general population (Castillo-Zacarías et al., 2021; Choi et al., 2020; Holmes et al., 2020; Melchor-Martínez et al., 2021). According to Díaz-Camal et al. (2022), the cost associated with antidepressant sales increased significantly in 2020, accounting for a 2- to 3-fold increase in the number of selective serotonin reuptake inhibitor (SSRI) prescriptions in Europe, North America, Asia, and Oceania. Among antidepressants, fluoxetine (FXT) is extensively used (Caiaffo et al., 2016).

Considering pharmaceutical antidepressant residues as water

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pollutants, sewage treatment plants are not efficient in removing many of these pollutants, and the detected environmental levels can represent a significant ecological risk to aquatic organisms (Castillo-Zacarías et al., 2021; de Souza et al., 2021; Ramírez-Morales et al., 2020). Safe discharges and water pollution prevention may involve measures such as ensuring the efficacy and quality of subsequent batches during pharmaceutical drug production. Preventing drug cross-contamination or adulteration requires specific measures, such as rigorous cleaning of processing facilities and equipment (Khan et al., 2020).

Various products can be applied in the cleaning process, such as one or more alkalinity or acidity sources, surfactants, chelating agents, and a solvent or water (Singh et al., 2022). Among them, surfactants represent a class of chemical compounds frequently used in various industrial and domestic applications (Siyal et al., 2020). Due to their high production volumes and consumption rates, these compounds are often detected in raw wastewater at high concentrations (Briels et al., 2023; Nunes and Teixeira, 2022). Luo et al. (2023) reported anionic surfactant concentrations as high as 23.17 mg L^{-1}. In municipal wastewater, levels range from 1.8 mg L^{-1} to 6.7 mg L^{-1} (Clara et al., 2007; Mondal et al., 2019), whereas industrial effluents have been found to contain concentrations exceeding 1000 mg L^{-1} (Kowalska et al., 2005; Orlandi et al., 2019; Zhang et al., 1999), highlighting the substantial load of surfactants discharged into these systems.

Sodium dodecyl benzene sulfonate (LAS) is an anionic surfactant widely used as a primary component in industrial cleaning products (Zhang et al., 2017). On the other hand, surfactants may induce adverse biological effects when disposed of in the aquatic environment. LAS: adverse morphological and hematological effects for zebrafish after 96 h of exposure (Libanio Reis Santos et al., 2024); *Daphnia similis* reproduction and development alterations (De Lima e Silva et al., 2022); hepatotoxicity was evidenced in bullfrog tadpoles (*Lithobates catesbeianus*), as well as morphometric, metabolic, and histopathological effects and changes in homeostasis (Franco-Belussi et al., 2021). It is essential to recognize that chemicals are often part of complex mixtures in the environment, and assessing the toxicity of these mixtures is challenging from both scientific and regulatory perspectives (Briels et al., 2023).

Persulfate-based advanced oxidation has been investigated as a viable and effective method for pollutant removal, widely applied in environmental remediation (Nunes et al., 2022; Song et al., 2019). Unlike traditional advanced oxidation processes, in which hydroxyl radicals (HO•) serve as the primary oxidants, the persulfate-based process involves the in situ generation of highly reactive and short-lived sulfate radicals (SO_4•$^-$) (Lee et al., 2020). Although persulfate (PS) is a potent oxidant, its activation is crucial in these processes, as persulfate alone has a weaker oxidation potential for removing organic pollutants (Ike et al., 2018). Therefore, various strategies can be applied for activation, including heat, transition metals (Fe^{2+}/Mn^{2+}), UV light, high temperature, high pH, and radiolysis (Nunes and Teixeira, 2022).

Ionizing radiation is an effective method for activating the degradation of emerging pollutants (Song et al., 2019). Regarding improvement in the degradation, the use of persulfate during the radiation process contributed to the degradation of emerging contaminants. The results showed that radiation can effectively activate persulfate, and hydroxyl radicals play a major role in the degradation process; however, their contribution to the degradation decreases as the absorbed dose increases (Su et al., 2023; Wang and Wang, 2018). EBI may require considerably low absorbed doses, offering high processing speed, a wide irradiation area, and also reducing the generation of secondary toxic intermediates (Ponomarev and Ershov, 2020). Several studies have demonstrated the application of EBI technology for the degradation and detoxification of contaminants, including pharmaceuticals (Silva et al., 2016; Truong et al., 2022), dyes (Garcia et al., 2020; Wang and Wang, 2022), and pesticides (Rodrigues et al., 2020).

Previous studies have shown that combining processes can enhance pollutant removal (Wang and Wang, 2022). The sulfate radical, produced as a supplementary reactive species, can enhance the degradation and mineralization of hazardous organic pollutants (Criquet and Leitner, 2011). Nevertheless, the increasing complexity of industrial and urban wastewater contaminants calls for a growing need for more efficient technologies. For instance, Fabbri et al. (2004) have reported a decrease in the photocatalytic degradation rate of 2,4,5-trichlorophenol pesticide in the presence of surfactant. In contrast, recent studies have found that surfactant and persulfate may enhance the oxidation of other pollutants in electrochemical processes (Lu et al., 2022; Escalona-Durán et al., 2020). Most studies focus on the use of EBI/PS for removing single components (Zhang et al., 2020; Bao et al., 2016; Roshani et al., 2011). Therefore, this study aimed to investigate the removal of the antidepressant fluoxetine in a mixture with LAS surfactant, using ionizing radiation and persulfate as a combined treatment process.

Initially, the study evaluated the effects of EBI on degradation and detoxification. Subsequently, the simultaneous effects of the initial persulfate concentration ($[PS]_0$) and absorbed doses were investigated and optimized using a Doehlert experimental design. Additionally, ecotoxicity assays were conducted to evaluate the reduction in toxicity after treatment. Electron beam treatment was also applied to the isolated surfactant in an aqueous solution to determine the removal efficiency and toxicity reduction. To our knowledge, none of these aspects has been discussed in the literature.

2. Materials and methods

2.1. Reagents

Sodium dodecylbenzene sulfonate surfactant [$CH_3(CH2)_{11}C_6H_4SO_3Na$; MM = 348.48 33 g mol^{-1}; CAS 25155-30-0, ≥99 %]; sodium persulfate ($Na_2S_2O_8$; ≥98 %) were obtained from Merck. Fluoxetine hydrochloride [$C_{17}H_{18}F_3NO.HCl$; MM = 309.33 g mol^{-1}; methyl [(3S)-3-phenyl-3-[4-(trifluoromethyl) phenoxy] propyl] amine]; CAS 54910-89-3; 98.8 %] was purchased from Divis Pharmaceuticals Pvt. Ltd. Acetonitrile and trifluoroacetic acid (TFA) were both HPLC-grade and obtained from Sigma-Aldrich (≥99.0 %). All the aqueous solutions used in the experiments were prepared using Milli-Q® water (18.2 MΩ cm).

2.2. Experiments

2.2.1. Irradiation procedure

Electron beam irradiation (EBI) was performed using a Dynamitron electron beam accelerator at 37.5 kW and 1.4 MeV. The experiments were performed on a batch scale. The aqueous solutions were placed in rectangular glass containers (Pyrex®) and irradiated, ensuring adequate and uniform electron beam penetration. The doses applied ranged from 1.0 to 5.0 kGy. The doses were confirmed using a Perspex Harwell Red dosimeter (Batch KZ-4034), and variations were lower than 5 %. A sample volume of 246 mL was employed, resulting in a maximum exposed liquid thickness of 4 mm, as reported in previous studies (Silva et al., 2016; Tominaga et al., 2021).

A Doehlert experimental design (Ferreira et al., 2004) was applied to investigate the optimal conditions for FXT removal in a mixture containing LAS surfactant by combining the EBI/PS process. Doehlert planning presents a structure that allows the scanning of a larger experimental region (Britto et al., 2021). The Doehlert design was adopted in this study due to its advantages, including a spherical and uniformly filled domain, comprehensive exploration of the experimental space, and the possibility of reusing experiments when extending the domain (Bensalah et al., 2010; Kiran et al., 2010). In addition, it enables the evaluation of interaction effects between variables while requiring fewer runs than Box–Behnken and Central Composite designs (Sautour et al., 2001). Another advantage is the flexibility to apply a different number of levels for each variable (Ferreira et al., 2004). In this study, the absorbed dose was evaluated at five levels, given its stronger

influence on the response, whereas persulfate concentration was tested at only three levels.

The effects of the absorbed dose (coded variable X_1) were evaluated at five levels (1.0, 2.0, 3.0, 4.0, and 5.0 kGy), while the initial PS concentration (coded variable X_2) was varied at three levels (0.5, 1.5, and 2.5 mM) (Table 1). Three replicates were conducted under central point conditions for statistical validation. In all experiments, the initial concentration of FXT was (5.22 ± 0.20) mg L^{-1}.

The coefficients of a second-order polynomial equation of the response surface model (Eq. (1)) were obtained by the least squares method using Statgraphics® and Matlab software, where y is the response variable and X_1 and X_2 correspond to the independent variables:

$$y = a + bX_1 + cX_2 + dX_1^2 + e\,X_2^2 + fX_1X_2 \quad (1)$$

2.2.2. Analytical method

The concentration of FXT was determined by high-performance liquid chromatography (HPLC) using a Shimadzu LC20 model, equipped with a RP18 column (250 mm × 4.6 mm, 5 μm) and a UV–vis detector (Shimadzu SPD20A model). The isocratic elution consisted of 35 % acetonitrile and 65 % TFA solution at a flow rate of 1.0 mL min^{-1}. The injection volume was 50 μL, and the detection wavelength was 230 nm. The FXT retention time was 20 min. For FXT, the calibration curve was determined, yielding an R^2 value = 0.9992, a limit of quantification (LOQ) = 0.013 mg $L^{-1,}$ and a limit of detection (LOD) = 0.041 mg L^{-1}.

The degradation of LAS surfactant after ionizing radiation was evaluated using the Methylene Blue Method with the Merck Spectroquant kit (Surfactants anionic Cell Test), followed by measurement on a UV–vis spectrophotometer. Samples containing a 10 mg L^{-1} surfactant solution were irradiated at doses of 2.5 and 5.0 kGy, as previously described. Samples were read at absorbance 652 nm, and the surfactant concentration result was obtained through a calibration curve ($y = 0.4088x + 0.1172$; $r^2 = 0.986$). This methodology is recommended by APHA 5540 C (APHA, 2005).

2.2.3. Ecotoxicity assays

Acute ecotoxicity tests were performed using *Daphnia similis* for individual compounds and the mixture (FXT + LAS), as well as before and after treatment with EBI/PS, under the highest FXT removal condition. The assays were conducted in accordance with ABNT Brazilian Standard NBR 12713 (ABNT, 2016). Neonates (6–24 h) were exposed to different concentrations of the samples (100 %, 50 %, 25 %, 12.5 %, and 6.25 %), with four replicates. For the control group, 20 neonates were used. The organisms were exposed to the samples for 48 h at 20 ± 1 °C. Physicochemical parameters were also assessed before and after the acute toxicity tests, including pH, dissolved oxygen (DO), and conductivity. At the end of 48 h, the number of immobile organisms in each sample was counted. The EC50-48h (expressed as a percentage) and its respective confidence interval were obtained using the Trimmed Spearman-Karber statistical test (Hamilton et al., 1977).

In addition, the toxicity of the binary mixtures was assessed using the toxic units (TUi) model (Eq. (2)), as described by Altenburger et al. (2003), Di Toro and McGrath (2000), and Ríos et al. (2017). The toxic unit of the binary mixture (TU_{mix}) corresponds to the sum of the TU_i of the individual compounds, and, for a binary mixture, TU_{mix} is given by Eq. (3):

$$TU_i = \frac{C_i}{EC_{50i}} \quad (2)$$

$$TU_{mix} = TU_A + TU_B \quad (3)$$

Where C_i represents the ratio between the concentration of a chemical in a mixture, $EC50_i$ is the acute toxicological endpoint, and TU_A and TU_B are the toxic units of the two chemicals in the mixture (A and B).

Table 1
– Doehlert experimental design: coded variables, real variables, and responses (% Fluoxetine Degradation). Different doses and $[persulfate]_0$ were evaluated for optimization. Aqueous solutions containing fluoxetine and surfactants were treated by the EBI/PS process. $[FXT]_0$ = (5.22 ± 0.20) mg L^{-1} and $[LAS]_0$ = (10.0 ± 0.46) mg L^{-1}.

Exp.	X_1: Dose (kGy)		X_2: $[PS]_0$ (mmol L^{-1})	
	Coded Values	Real values	Coded values	Real values
1	0.0	3.00	0.0	1.50
2	1.0	5.00	0.0	1.50
3	0.5	4.00	0.866	2.50
4	−1.0	1.00	0.0	1.50
5	−0.5	2.00	−0.866	0.50
6	−0.5	2.00	0.866	2.50
7	0.5	4.00	−0.866	0.50

3. Results and discussion

3.1. Toxicity assessment of the single and mixture effects

The acute toxicity effects of FXT, the anionic surfactant LAS, and their mixture to *Daphnia similis* were summarized in Table 2. The results indicated greater toxicity of FXT ($EC50_{48h}$ = 1.45 mg L^{-1}) compared to LAS ($EC50_{48h}$ = 7.23 mg L^{-1}). Acute toxicity assays were also carried out with PS, showing $EC50_{48h}$ values of 443 mg L^{-1} (1.86 mmol L^{-1} PS).

Previous studies have demonstrated that low concentrations of FXT can impact the reproduction of *Daphnia similis* (LOEC = 0.40 mg L^{-1}) after 21 days of exposure (Tominaga et al., 2022). Ding et al. (2017) reported that FXT can accumulate and induce physiological (an increase in filtration rate) and biochemical disturbances (neurotoxicity) in *D. magna* at concentrations of 0.5 and 5 μg L^{-1}. Regarding the surfactant, Wang et al. (2009) estimated an EC50 of 7.5 mg L^{-1} for *Daphnia magna* after 24 h of exposure.

The toxicity of binary mixtures (FXT + LAS) was determined using the toxic unit model. The TU_{mix} suggests that the concentration addition model probably best explains the mode of action of the mixture (TU_{mix} = 1.0 ± 0.2). Furthermore, the presence of antagonist effects can be observed ($TU_{mix} > 1.0$). There is a lack of information on the toxicity of surfactants and other organic pollutants when mixed. Therefore, further studies should be conducted under different conditions, as previous research has indicated that both antagonistic and synergistic effects can be observed, depending on the concentration of pollutants in the mixture (Tominaga et al., 2022).

3.2. Effect of EBI on the degradation and toxicity of mixtures and application of combined EBI/PS

Wastewater treatment requires time, energy, and cost. Therefore, it is crucial to optimize processes as a key goal of sustainable development to reduce energy consumption and improve strategic processes globally. Test design can enhance the production process, resulting in higher quality, increased efficiency, and greater reliability (Shojaei and Shojaei, 2021). The initial working concentrations of FXT and LAS were 5.22 ± 0.20 and 10.74 ± 0.21, respectively, selected within the range of values from the literature for pharmaceuticals (Ashfaq et al., 2017; Hussain et al., 2016) and surfactants (Zhang et al., 1999; Orlandi et al., 2019) in real contaminated aqueous matrices.

Initial experiments, conducted without the addition of persulfate,

Table 2
– *Daphnia similis*-EC50 values, for mixtures of FXT and LAS.

FXT (A) EC50 mg L^{-1}	LAS (B) EC50 mg L^{-1}	Mixture EC50 mg L^{-1}	TU_A	TU_B	TU_{mix}
1.45 ± 0.36 [a]	7.23 ± 0.74	2.97 ± 0.67	1.02	0.21	1.23

[a] Tominaga et al. (2021).

were performed at the highest absorbed dose in this study (5.0 kGy) to evaluate maximum degradation at the higher dose. The results indicated efficient removal of 90.1 ± 1.1 % for FXT. Then, a Doehlert experimental design was carried out to evaluate the simultaneous effect of the initial persulfate concentration ($[PS]_0$) and absorbed dose on FXT degradation, and to optimize the better experimental condition.

The main results are summarized in Table 3. The ANOVA test indicated that the effects of absorbed dose and $[PS]_0$ were statistically significant (F test with p-value <0.05), as shown by the Pareto chart. The coefficient of determination of the corresponding response surface model was $R^2 = 0.988$ (Table S1 and Fig. S1).

The effects estimated from the Doehlert design were represented in the Pareto chart (Fig. S1). The dose had a significant positive effect on FXT degradation in the EBI/PS process, indicating that higher doses resulted in increased removal efficiency. However, the dose effect was followed by a negative quadratic term of the dose, which indicated that an increase in the initial persulfate concentration above an optimal concentration resulted in process inhibition. Then, the effect was followed by an interaction between dose and initial persulfate concentration. Lastly, the linear quadratic term of persulfate initial concentration presented the lowest effect. These findings demonstrate that dose exerts the predominant influence on the system, being the most significant variable under the experimental conditions evaluated. Additionally, the results showed that at a lower initial $[PS]_0$, an increase in dose resulted in greater FXT removal, achieving 96.2 % at $[PS]_0 = 0.5$ mmol L^{-1} and dose = 4.0 kGy, while at higher $[PS]_0$, an increase in the dose resulted in a reduction in FXT degradation (Fig. 1).

The fit by the polynomial model, the response surface, and the corresponding counter plot are shown in Eq. (4) and Fig. 1.

$$\%\ \text{Degradation} = 95.6695 + 2.82874X_1 + 0.56312X_2 - 2.46634X_1^2 - 1.19808X_2^2 - 2.00315 \times 1 \times {}_2 \quad (4)$$

EBI leads to water radiolysis, which generates oxidative radicals (hydroxyl radicals, •OH) and reductive species (hydrogen atoms, H•, and solvated electrons, e_{aq}^-) that result in the rapid degradation of organic pollutants (Cooper et al., 2004), as summarized by Eq. (5) with yields (G-values = number of moles formed or decomposed per Joule, absorbed energy) expressed in μmol J^{-1} at pH 7.

$$H_2O \rightarrow [0.28]\ HO\bullet + [0.06]\ H\bullet + [0.27]\ e_{aq}^- + [0.05]\ H_2 + [0.27]\ H_3O + {}_{aq} + [0.07]\ H_2O_2 \quad (5)$$

The obtained results showed an improvement in FXT degradation with increasing dose, which can be attributed to the higher generation of reactive radicals (Fig. 1). The improvement in FXT removal results from the increased production of the oxidative sulfate radical ($SO4^{\bullet-}$), formed from the reaction between persulfate anions and solvated electrons (Eq. (6)) (Criquet and Leitner, 2011; Neta et al., 1988). In the presence of PS, a comparable removal efficiency was obtained at lower doses (90.6 % FXT removal with 1.0 kGy and 1.50 mmol L^{-1} PS), when compared to EBI alone at the highest applied dose (90.1 ± 1.1 % at 5.0 kGy).

Table 3
– Doehlert experimental design: real variables and responses (% Fluoxetine Degradation). Different doses and $[persulfate]_0$ were evaluated for optimization. Aqueous solutions containing fluoxetine and surfactants were treated by the EBI/PS process. $[FXT]_0 = (5.22 \pm 0.20)$ mg L^{-1} and $[LAS]_0 = (10.0 \pm 0.46)$ mg L^{-1}.

Exp.	X_1: Dose (kGy)	X_2: $[PS]_0$ (mmol L^{-1})	% Degradation
1	3.00	1.50	95.7[a]
2	5.00	1.50	95.8
3	4.00	2.50	95.4
4	1.00	1.50	90.6
5	2.00	0.50	91.2
6	2.00	2.50	93.9
7	4.00	0.50	96.2

[a] Performed in triplicate, giving (95.7 ± 0.2) %.

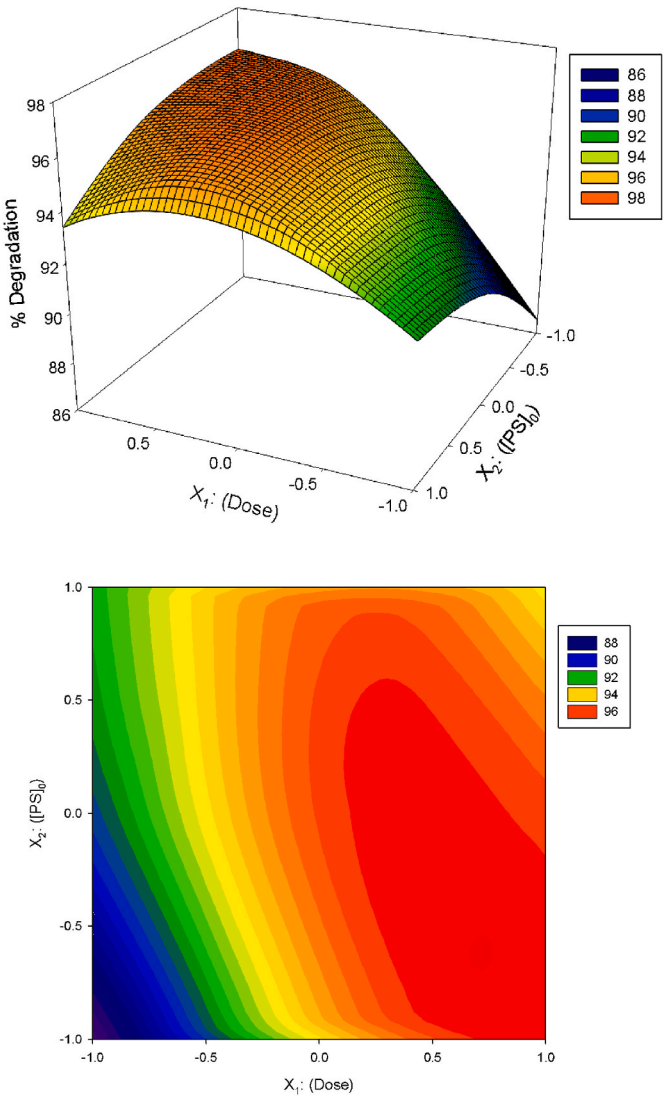

Fig. 1. – Response surface and contour plot for % FXT removal varying doses from 1.0 to 5.0 kGy and $[PS]_0$ from 0.5 to 2.5 mmol L^{-1} $[FXT]_0 = 5.88 \pm 0.05$ mg L^{-1}, $[LAS]_0 = 10.76 \pm 0.26$ mg L^{-1}.

In addition, persulfate can also react with H• to produce sulfate radicals (Eq. (7)). However, H• production is not as significant as the production of solvated electron (G (H•) = 0.06 and $G(e_{aq}^-) = 0.27$), and H• is much less reactive ($k = 2.5 \times 10^7$ L $mol^{-1}s^{-1}$) (Criquet and Leitner, 2011). Moreover, persulfate also reacts with and •OH radicals to generate $S_2O_8^{\bullet-}$ radicals (Eq. (8)).

$$S_2O_8^{2-} + e_{aq}^- \rightarrow SO_{4-} + SO_4^{2-} \quad k = 1.1 \times 10^{10}\ L\ mol^{-1}s^{-1} \quad (6)$$

$$S_2O_8^{2-} + H\cdot \rightarrow SO_{4-} + H^+ + SO_4^{2-} \quad k = 2.5 \times 10^7 L\ mol^{-1}s^{-1} \quad (7)$$

$$S_2O_8^{2-} + \cdot OH \rightarrow OH^- + S_2O_{8-} \quad k = 8 \times 10^4 L\ mol^{-1}s^{-1} \quad (8)$$

However, the response surface and contour plots indicate that beyond a specific dose, degradation decreases (Fig. 1), likely due to radical recombination. When persulfate (PS) is present in excess in the system, it can react with OH• and H• radicals generated through the recombination of excess $SO_4\bullet^-$, which reduces the overall efficiency of the process (eqs. (9)–(11)) (Senthilkumar et al., 2022).

$$SO_4^{\bullet-} + SO_4^{\bullet-} \rightarrow S_2O_8^{2-} \quad (9)$$

$$SO_4^{\bullet -} + S_2O_8^{2-} \rightarrow SO_4^{2-} + S_2O_8^{\bullet 2-} \quad (10)$$

$$S_2O_8^{2-} + {}^{\bullet}OH \rightarrow S_2O_8^{\bullet -} + OH^- \quad (11)$$

The results obtained herein showed a reduction in efficiency when the persulfate concentration was increased from 1.5 to 2 mmol L^{-1}. Low degradation, ranging from 1.5 to 2.0 mmol L^{-1}, has also been reported in the combined gamma/PS process for removing triclosan and trimethoprim due to an excess of persulfate (Zhang et al., 2016, 2021). Similarly, increasing persulfate (PS) concentration led to lower degradation, consistent with the scavenging effect of excessive $S_2O_8^{2-}$, which reduces the availability of reactive radicals, as previously reported (Nunes et al., 2022).

In addition, the adverse effects observed for the quadratic terms (AA and BB) and the interaction term (AB) suggest that the system does not respond linearly to simultaneous increases in dose and PS concentration. These effects indicate the presence of optimal conditions, beyond which the process's efficiency is hindered. This behavior underscores the importance of striking a balance between oxidant dose and persulfate concentration to optimize degradation efficiency while minimizing radical scavenging and side reactions.

Experiments were carried out with the anionic LAS surfactant, pure LAS (Initial concentration: 10 mg L^{-1}), and irradiations were performed to evaluate the efficiency of the process in degrading and reducing the toxicity of this compound, which is present in several effluents and aquatic matrices. The data obtained were summarized in Table 4. Electron beam irradiation resulted in a significant degradation of the LAS surfactant, with an increase in the dose used, achieving an efficiency higher than 90 %. The initial concentration was reduced to 0.78 mg L^{-1} ± 0.08 (2.5 kGy) and 0.55 mg L^{-1} ± 0.05 (5.0 kGy). Regarding toxicity, a 56.06 % reduction was achieved at 2.5 kGy. By increasing the absorbed dose to 5.0 kGy, a 73.39 % reduction in toxicity was measured.

The results obtained herein were promising for treating LAS surfactant with an electron beam, as irradiation significantly reduced toxicity and surfactant concentration. Similar results were obtained by Romanelli et al. (2004), with an 88 % reduction in the acute effects of *D. similis* and degradation exceeding 85 % at a 3.0 kGy dose. Recently, Jiao et al. (2022) obtained degradation of 50.9 %–89.1 % under 2.0 kGy and 10 kGy, respectively.

Regarding toxicity, after the combined treatment, acute toxicity assays were conducted under the optimal FXT removal conditions (1.1 mmol L^{-1} PS and 4.5 kGy). The results indicated an increase from 3.36 TU to 5.85 TU, possibly due to more toxic byproducts related to persulfate degradation. Previous studies have demonstrated a reduction in FXT toxicity following EBI treatment, suggesting the formation of less toxic byproducts (Silva et al., 2016; Tominaga et al., 2021). The herein increase might be related to the formation of more toxic byproducts related to persulfate degradation. A previous study also found increased toxicity to aquatic organisms (*Daphnia similis* and *Vibrio fischeri*) after EBI treatment with persulfate (Tominaga et al., 2024). In the study, an increase in metformin removal was reported after PS addition. Nevertheless, an increase in toxicity was also reported. These results reinforce the importance of ecotoxicity assays.

Additionally, the scalability of electron beam irradiation, coupled with other technologies, is a crucial consideration for its practical application in real-world wastewater treatment. While laboratory studies demonstrate its strong oxidative capacity and effectiveness in degrading a wide range of contaminants (Saien et al., 2023; Nunes et al., 2022; Görmez et al., 2020), translation to full-scale operations requires careful evaluation of several key factors.

Table 4
– LAS degradation and toxicity assessment with *D. similis* after electron beam irradiation. $[LAS]_0 = (10.0 \pm 0.46)$ mg L^{-1}.

Doses (kGy)	LAS degradation (%)	Toxicity reduction (%)
2.5	92.2 ± 0.84	56.06 ± 1.69
5.0	94.5 ± 0.56	73.39 ± 1.3

The electron beam irradiation process is a highly economical and sustainable technology, as it does not require the addition of catalysts, activators, or other additives, and a relatively pure final product may be obtained (Hossain et al., 2018). The practical applications of this technology have already been reported by Ponomarev and Ershov (2020), Hossain et al. (2018), and Han et al. (2012). Wang et al. (2022) demonstrated a practical application of electron beam (EB) irradiation with Fenton oxidation to enhance the treatment of dyeing wastewater. According to them, combining Fenton oxidation and EBI as a second step enhances radical production, improving pollutant removal and mineralization.

Laboratory and pilot-scale studies confirmed the efficiency and scalability of the EBI/Fenton system for industrial wastewater treatment. Furthermore, Hossain et al. (2018) estimated a total cost (including operation, capacity, and water discharge) for electron beam treatment of US$0.29/$m^3$ for 10,000 m^3/day, and the cost value decreases with increasing treated volume (US$0.041/$m^3$ for treatment of 200,000 m^3/day).

Ionizing radiation with persulfate addition resulted in increased mineralization and the removal of various pollutants (Bao et al., 2016; Criquet and Leitner, 2011, 2012; Roshani and Leitner, 2011; Roshani and Vel Leitner, 2011; Zhang et al., 2016, 2020, 2021). In the literature, several advanced oxidation processes have been proposed for FXT removal, as described in Table 4. Generally, Advanced Oxidative Processes can be effective for removing fluoxetine. Nevertheless, most studies focus on the degradation of single compounds. The decrease in the degradation rate of (2,4,5-trichlorophenol) in the presence of surfactant (sodium dodecyl sulfate) was carried out by Fabbri et al. (2004).

According to Wang and Wang (2021), significant changes in the toxicity of the treated wastewater can be found (increase or decrease). Although our results indicated that the addition of persulfate before EBI increased fluoxetine removal, previous studies have shown that EBI/PS can act as a pre-treatment for biological processes. Increased biodegradability after gamma radiation/persulfate treatment was noted by Bao et al. (2016) and Z. Zhang et al. (2020). Nevertheless, there remains a lack of studies examining the mixture and toxicity assessment of intermediate products generated after pollutant treatment techniques used to mitigate the impact of pollutants in the aquatic environment, as shown in Table 5.

4. Conclusion

Electron beam irradiation was effective in removing fluoxetine from a binary mixture of fluoxetine and sodium dodecyl benzene sulfonate (5.22 ± 0.20 mg L^{-1} fluoxetine and 10.74 ± 0.21 mg L^{-1} LAS), achieving a degradation of 90.1 ± 1.1 % at 5.0 kGy. The combined process (EBI/PS) increased FXT removal, even at low doses, with a 90.6 % removal at 1.0 kGy ($[PS]_0 = 1.5$ mmol L^{-1}). The highest degradation value achieved was 96.2 % ($[PS]_0 = 1.5$ mmol L^{-1} and four kGy). These results highlight the crucial role of experimental design in optimizing operational conditions, since our results also showed that a high concentration of persulfate negatively affected fluoxetine removal efficiency due to inefficient conversion into $SO_4\bullet-$ radicals. The toxicity tests carried out under the best-optimized condition ($[PS]_0 = 1.1$ mmol L^{-1} and 4.5 kGy) indicated an increase in toxicity to *Daphnia similis*, suggesting the formation of more toxic by-products. These results indicate that a multidisciplinary approach is required to evaluate the impacts of the generated by-products, long-term effects of the treated water, and explore different matrices.

CRediT authorship contribution statement

Flávio Kiyoshi Tominaga: Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization. **Roberta Frinhani Nunes:** Methodology, Formal analysis. **Vanessa Silva Granadeiro Garcia:** Writing – review & editing, Methodology, Formal analysis.

Table 5
Comparison of the removal efficiency of the antidepressant fluoxetine using different advanced oxidation processes.

Process	Conditions	% Removal	Toxicity	Reference
Photo-Fenton	Citrate complex (Fecit) = 1 μM [H_2O_2]: 50 μM pH = 4.5 $[FXT]_0 = 100$ μg L^{-1} (single aqueous solution, distilled water)	80 % removal after 20 min	–	Perini et al. (2017).
UV irradiation	High-pressure mercury lamp (300 W). Temperature = 20 ± 2 °C. $[FXT]_0 = 1$ mg L^{-1} (single aqueous solution, fresh egg water)	97.6 % ± 0.25 after 60 min	Both untreated and treated samples affected zebrafish development (Hatching rate, body length, and heart rate)	Pan et al. (2022)
Hybrid catalytic/ ozonation	High-pressure Hg lamp (75 W) TiO_2 (0.050 g L^{-1}) pH 11 $[FXT]_0 = 34$ mg L^{-1} (single aqueous solution, deionized water)	Complete removal and 50 % mineralization at 60 min		Méndez-Arriaga et al. (2011)
	High-pressure Hg lamp (75 W) TiO_2 (0.050 g L^{-1}) pH 11 $[H_2O_2]_0 = 0.12$ mmol L^{-1} $[FXT]_0 = 34$ mg L^{-1} (single aqueous solution, deionized water)	Complete removal and Mineralization further to >70 % at 60 min		
	UV/O_3 $[FXT]_0 = 34$ mg L^{-1} (single aqueous solution, deionized water)	100 % in the first 10 min		
	$[O_3]_0 = 25$ mg L^{-1} pH 11 $[FXT]_0 = 34$ mg L^{-1} (single aqueous solution, deionized water)	100 % removal after 10 min		
Ozone/H_2O_2	$[O_3]_0 = 30$ mg L^{-1}, $[H_2O_2]_0 = 0.02$ mmol L^{-1} $[FXT]_0 = 50$ mg L^{-1}	86.14 % removal after 20 min	-	Aghaeinejad-Meybodi et al. (2015)
Ozonation/nano-γ-alumina	$[O]_0 = 30$ mg L^{-1} [Catalyst] = 1 g L^{-1} [FXT]0 = 28.56 mg L^{-1} (single aqueous solution, deionized water)	96.14 % removal in minutes of reaction	–	Aghaeinejad-Meybodi et al. (2019)
Electron beam irradiation (EBI)	Dynamitron® electron beam accelerator (37.5 kW and 1.4 MeV). $[FXT]_0 = 10$ mg L^{-1} (single aqueous solution,ultra-pure water)	>98.0 % at 5.0 kGy	60 % toxicity removal for *Daphnia similis*	Tominaga et al. (2021)
EBI/PS	($[PS]_0 = 1.1$ mmol L-1; absorbed dose of 4.5 kGy (presence of LAS,ultra-pure water)	95.7 4.5 kGy	Increase in toxicity for *Daphnia similis*	This work

Antonio Carlos Silva Costa Teixeira: Writing – review & editing, Supervision. **Sueli Ivone Borrely:** Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.radphyschem.2025.113550.

Data availability

Data will be made available on request.

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