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Electron Beam Irradiation-Induced Degradation of Sulfadiazine in Aqueous Solutions

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Abstract: The degradation of sulfadiazine (4-amino-*N*-pyrimidin-2-yl-benzenesulfonamide, SDZ), a widely used sulfonamide antibiotic, in aqueous solution under electron beam irradiation was investigated to explore its potential as an Advanced Oxidation Process for environmental remediation. This study evaluated the effects of irradiation dose, initial sulfadiazine concentration, and initial pH on the degradation efficiency. It was found at 0.5 kGy that the degradation efficiency decreased with increasing initial SDZ concentration, from 83.0% at 5 mg/L to 35.0% at 30 mg/L. The kinetic results showed a pseudo-first order model. The degradation efficiencies of 30 mg/L SDZ reached 80.8%, 75.3%, 69.5% and 69.8%, respectively, at pH 3.0, 6.3, 9.0, and 11.0 at 3.0 kGy, indicating the pH dependence to SDZ degradation under electron beam. The maximum removal efficiency was around 90% after UV analysis and 99% after HPLC analysis for 10mg/L SDZ at absorbed doses of 2–3 kGy and pH 6.3. Increasing the degradation efficiency of 10 mg/L SDZ from 0.5 kGy to 3.0 kGy showed the dose dependence on SDZ removal. Reactive species generated during irradiation, including hydroxyl radicals, hydrogen radicals, and solvated electrons, were identified as primary contributors to the degradation process. The effect of reactive species on the degradation of 10 mg/L SDZ was evaluated at variable doses, revealing the following trend: $\bullet OH > \bullet H > e_{aq}^-$. Transformation products were characterized using high-performance liquid chromatography (HPLC) and mass spectrometry (MS), providing insights into the degradation pathway. The results demonstrate that electron beam irradiation is an effective and sustainable method for sulfadiazine removal in water treatment systems, offering an innovative approach to mitigating antibiotic pollution in aquatic environments.

Keywords: electron beam; sulfadiazine; degradation; mechanism



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1. Introduction

Sulfonamides are among the most commonly used antibacterial agents in the world for veterinary and human medicine, especially in animal husbandry and fish farms, due to their affordability, minimal toxicity, and high effectiveness against common bacterial infections [1,2]. Studies have shown that sulfonamides rank as the second most commonly used veterinary antibiotics in the European Union, representing approximately 21% of sales in the UK and ranging between 11% and 23% in various other European nations [3]. After use, their high solubility and rapid excretion rates in river basins and catchment areas make them likely contaminants of environmental water systems and they can cause antibiotic

resistance [4]. Sulfadiazine (SDZ) belongs to the sulfonamide class of antibiotics frequently detected in high concentrations (502.43 ng L^{-1}) in surface water and $34\text{--}859 \text{ ng L}^{-1}$ in the treated effluent [5]. A primary pathway for its introduction into the environment is through the excretion of urine and feces from treated livestock and poultry. When manure containing SDZ is applied as a fertilizer, the antibiotic can infiltrate agricultural soils, potentially entering the food chain through plant absorption. Additionally, SDZ may leach into groundwater or be transported to surface water through runoff, ultimately contributing to antibiotic resistance and posing toxicological risks to ecosystems and human health [3,6].

Traditional wastewater treatment methods for industrial and municipal effluents are less sufficient in removing antibiotic contaminants because of their toxicity and bacterial resistance [7]. With the aim of limiting the harmful effects of these various types of refractory contaminants in the environment, effective and eco-friendly treatment strategies have been developed such as Advanced Oxidation Processes (AOPs). Such techniques are based on the in situ generation of hydroxyl radicals ($\bullet\text{OH}$), which are among the most powerful oxidizing species available for industrial wastewater treatment [8]. Radiation technology, recognized as a highly effective and distinctive Advanced Oxidation Process, has been gaining increasing attention in recent years. Electron beam irradiation (EBI) is capable of breaking down organic pollutants in wastewater through two primary mechanisms: direct interaction with the high-energy beam and indirect degradation by reactive species (hydroxyl radicals, hydrated electrons, and hydrogen atoms) [9] produced during water radiolysis [10,11]. Extensive research has demonstrated its effectiveness in removing organic contaminants, eliminating bacteria, and reducing antibiotic resistance genes [12–16]. Moreover, EBI offers high processing efficiency, reducing treatment time and costs, while being capable of decomposing and treating multiple types of pollutants with no secondary contamination making it cleaner than traditional AOPs like Fenton or photocatalysis and has a proven track record of safe use in food, seafood, and other sterilization applications, even at high pollutant concentrations [17]. However, its high initial cost, energy demands, and infrastructure requirements limit its widespread adoption compared to photocatalysis, electrocatalysis, or sonication, which are more accessible and cost-effective. EB irradiation is promising for large-scale wastewater treatment.

Despite its potential, limited research has explored the use of EBI for sulfadiazine degradation, and further investigation is needed to understand the key parameters influencing the process. While Zhu et al. [17] demonstrated the effectiveness of e-beam irradiation for SDZ degradation; their study lacked a detailed analysis of reactive species' contributions and the influence of pH across a broad range. Similarly, Da Wang et al. (2024) [18] focused on TiO_2 -based photocatalysis, which is limited by catalyst recovery and energy requirements. This study addresses these gaps by evaluating the efficacy of EBI in degrading SDZ in aqueous solutions by systematically examining various factors that affect the degradation process. The effect of radiation dose will be assessed to determine its influence on degradation efficiency and reaction kinetics. Additionally, the impact of initial SDZ concentration will be analyzed to understand its role in degradation behavior and rate constants. Another critical aspect of this study is the variation in pH across a broader pH range (3–11) providing a more comprehensive understanding of pH effects, as it plays a significant role in altering the dominant reactive species involved in the degradation process. To further elucidate the mechanism, the contribution of different reactive species will be investigated using radical scavengers to identify their specific roles and hierarchy in SDZ degradation. Furthermore, the study will focus on identifying intermediate degradation products through liquid chromatography–mass spectrometry (LC/MS), providing insight into the degradation pathways and assessing the potential toxicity of the byproducts formed.

By addressing these aspects, this research provides a comprehensive assessment of EBI as a viable method for the removal of SDZ from contaminated water sources. The study contributes to the broader application of radiation-based technologies in environmental remediation and wastewater treatment, offering valuable insights into the optimization and scalability of EBI for effective pollutant degradation.

2. Materials and Methods

2.1. Chemicals

Sulfadiazine (>99%), acetonitrile HPLC gradient grade ($\geq 99.9\%$), tert-butanol ($\geq 99.5\%$), potassium dichromate (99%), silver nitrate (99%), formic acid ($\geq 96\%$), and perchloric acid (95%) were purchased from Sigma Aldrich (Hamburg, Germany). Distilled water for dilutions was obtained from the Thermo-Fischer distillation unit from Merck (Darmstadt, Germany). Dinitrogen oxide gas (98%) and argon gas 5.0 were obtained from Air Products Sp. Z.o.o. (Siewierz, Poland). Hydrochloric acid (35%) and sodium hydroxide ($\geq 98\%$) obtained from Sigma Aldrich (Hamburg, Germany) were used for pH adjustment.

2.2. SDZ Degradation

SDZ degradation was performed using an electron beam ILU6 accelerator at 1.65 MeV, a pulse frequency of 2 Hz, a pulse duration of 400 μ s, and a current of 50 mA located at the Institute of Nuclear Chemistry and Technology (INCT, Warsaw, Poland). The absorbed dose was measured with a low-range dichromate dosimeter, consisting of 0.5 mM silver dichromate in 0.1 M aqueous perchloric acid [19], which was prepared by dissolving 0.5 mM potassium dichromate and 1 mM silver nitrate. Low-density polyethylene (LDPE) sleeve bags were used as the reaction vessels, with 30 mL of each SDZ solution placed inside. The bags were then irradiated at different doses (0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 kGy). The samples were irradiated at a total absorbed dose of 3.0 kGy, delivered in six equal fractions of 0.5 kGy each (6×0.5 kGy). Each fraction was administered separately, ensuring uniform exposure and allowing potential observation of intermediate degradation effects between irradiations. The corresponding irradiation time for each dose fraction was determined by the beam parameters, with each 0.5 kGy dose requiring an exposure time of 12.5 s.

In order to investigate the effect of pH on the degradation of sulfadiazine (SDZ), solutions of 10 mg/L SDZ were prepared and adjusted to the desired pH using 0.1 M HCl and 0.1 M NaOH. The selected pH values for the study were 3.0, 6.3, 9.0, and 11.0. The solutions were then subjected to electron beam irradiation at varying doses of 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 kGy to evaluate the extent of degradation.

To assess the role of different reactive species in the degradation process, specific scavengers were employed to selectively quench hydroxyl radicals, solvated electrons, and hydrogen radicals. Nitrous oxide (N_2O) was used to remove solvated electrons, while 0.5 M tert-butanol in the presence of argon (Ar) was used to scavenge hydroxyl radicals [20]. Additionally, 0.5 M of tert-butanol in an argon-saturated environment at pH 2 was used to study the effect of hydrogen radicals [21]. Before irradiation, N_2O and Ar were bubbled into the solutions for 30 min to ensure the removal of dissolved oxygen and the establishment of the desired reactive species environment.

2.3. Analytical Method

The UV–Vis spectral analysis and removal efficiency of SDZ were measured on a JASCO V-670 spectrophotometer (Warsaw, Poland) over a wavelength range of 190–400 nm with a maximum detection wavelength of 265 nm.

SDZ's concentration to examine the effect of reactive species was determined with a Shimadzu class vp high-performance liquid chromatography (HPLC) (Agilent 1200,

Agilent, Santa Clara, CA 95051, USA) and a diode array detector (DAD). The column was a Gemini 5 μm (Phenomenex, Warsaw, Poland) C18 110 \AA 250 \times 4.6 mm operated at an oven temperature of 40 $^{\circ}\text{C}$, flow rate of 1 mL/min, and the injection volume was 20 μL . A mixture of acetonitrile and distilled water with a ratio of 50:50 (v/v) was used as the mobile phase.

LC-MS (Q Exactive, Thermo Scientific, Tianjing, China) enabled the identification of byproducts formed under EB. LC-MS was performed on an Agilent 1200, USA, equipped with a C18 column (5 mm, 4.6 \times 150 mm) and a diode array detector (DAD). The measurement wavelength was set at 275 nm and the column temperature at 30 $^{\circ}\text{C}$. The flow rate was maintained at 0.2 mL min^{-1} . The initial mobile phase consisted of 90% water (0.1% formic acid) (A) and 10% acetonitrile (B). The level of solvent B increased to 100% within 30 min, maintained for 2 min, and returned to initial settings in 2 min.

The pH of the samples was measured using an Elmetron CX-461 multimeter (Zabrze, Poland).

3. Results and Discussions

3.1. Dose Influence on SDZ Degradation

Figure 1 presents UV absorption spectra of 10 mg/L SDZ before and after irradiation under EB at variable doses (0, 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 kGy). It was noticed that the absorbance of SDZ decreases with an increase in dose at the wavelength of 264 nm. At low doses, that is from 0.5 to 1.0 kGy, a gradual reduction in absorbance was observed, indicating partial degradation while at intermediate doses (1.5–2.0 kGy) a more significant decrease in absorbance described a consistent structural modification of SDZ. By 2.5 to 3.0 kGy, the absorbance had diminished by approximately 90%, implying near-total degradation of SDZ. This trend suggests that the degradation of SDZ under EB is dose-dependent. Previous research on various compounds has reported the same trend [22–24].

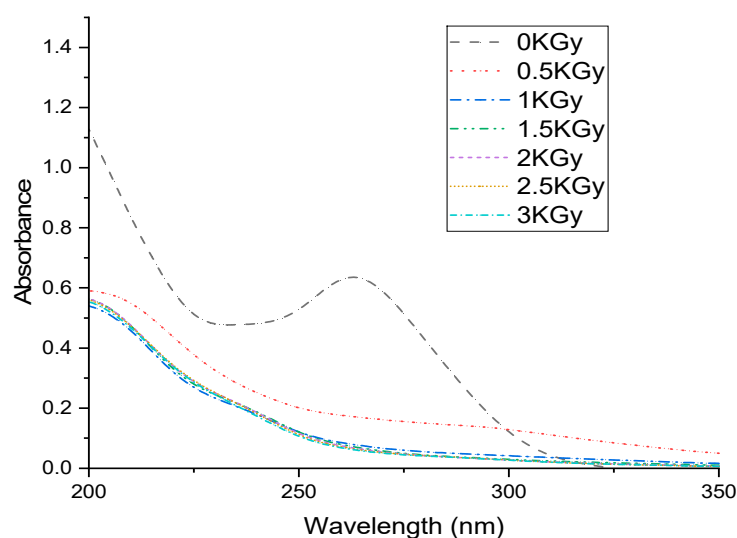
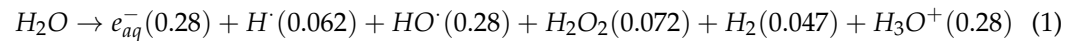


Figure 1. UV absorption spectra of 10 mg/L SDZ (pH = 6.3; dose: 0–3 kGy, $\lambda = 264$ nm).

3.2. Effect of SDZ Initial Concentration Under EBI

The degradation of organic contaminants by EB irradiation is induced by the radical species formed through water radiolysis, as shown in Equation (1). The value in brackets represents the radiochemical yield of the reactive species, known as the G value. It is

defined as the number of molecules, atoms, or radicals generated or consumed in water per joule of absorbed energy (μmolj^{-1}) [17].



The degradation of SDZ under EB was investigated at varying doses of 0.5, 1, 1.5, 2, 2.5, and 3 kGy across different initial concentrations of 5, 10, 15, 20, and 30 mg/L. As shown in Figure 2, the removal efficiency consistently decreased as the SDZ concentration increased at a dose of 0.5 kGy. The removal efficiencies observed on Figure 2 were 83.4%, 72.2%, 64.2%, 52.1%, and 35.3% for SDZ concentrations of 5, 10, 15, 20, and 30 mg/L, respectively. This result is in line with previous studies, which asserts that degradation decreases as the concentration of antibiotics increases [25,26].

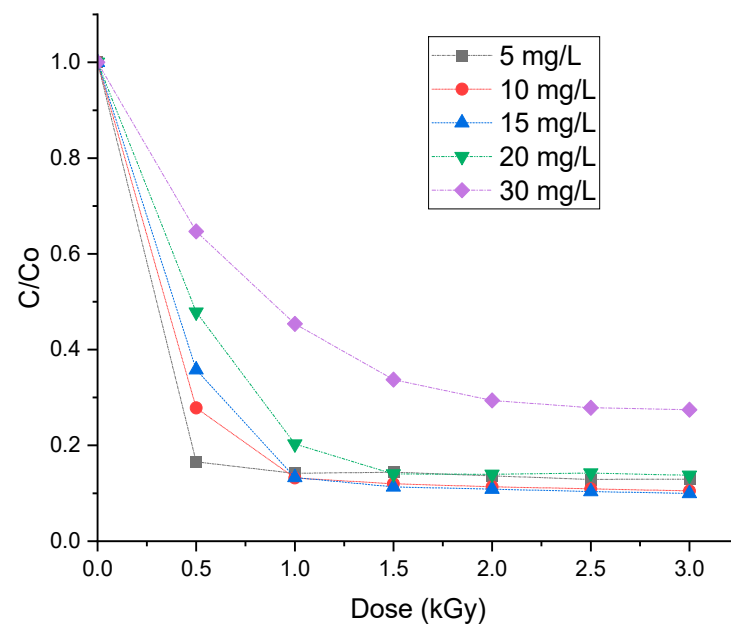


Figure 2. Degradation of SDZ monitored by UV/Vis spectroscopy at different initial concentrations (pH = 6.3; dose: 0–3 kGy).

The rate of SDZ removal with respect to dose followed the pseudo-first order kinetic described by Equation (2)

$$-\ln\left(\frac{C}{C_0}\right) = kD \quad (2)$$

where C_0 is the initial concentration of SDZ (mg/L); C is the concentration of SDZ after radiation (mg/L); D represents the absorbed dose (kGy); and k stands for the dose constant (kGy^{-1}).

The dose constant (k) values for SDZ degradation under electron beam irradiation were determined at various initial concentrations of 5, 10, 15, 20, and 30 mg/L, yielding values of 1.65, 1.67, 1.66, 1.40, and 0.75 kGy^{-1} , respectively (Figure 3). The observed decrease in k with increasing SDZ concentration suggests that at higher concentrations, the interaction between SDZ molecules and reactive species becomes less efficient. This could be attributed to the limited availability of active radicals relative to the increased SDZ molecules in solution. Moreover, high concentrations of the parent compound may have resulted in the formation of more degradation byproducts. This, in turn, creates competition between SDZ and its byproducts for reactions with reactive radicals, leaving higher residual SDZ levels [27]. A similar trend was reported by Liu et al. [28] for SDZ degradation using gamma irradiation, where the pseudo-first-order rate constant decreased

from 1.04 to 0.76 kGy⁻¹ as the initial SDZ concentration increased from 10 to 30 mg/L. These comparative results demonstrate that while both radiation-based methods show concentration-dependent efficiency, electron beam irradiation exhibits marginally faster kinetics, likely due to its higher instantaneous radical production. The consistent decrease in *k* values across both processes confirms the fundamental limitation of radical availability at higher contaminant concentrations.

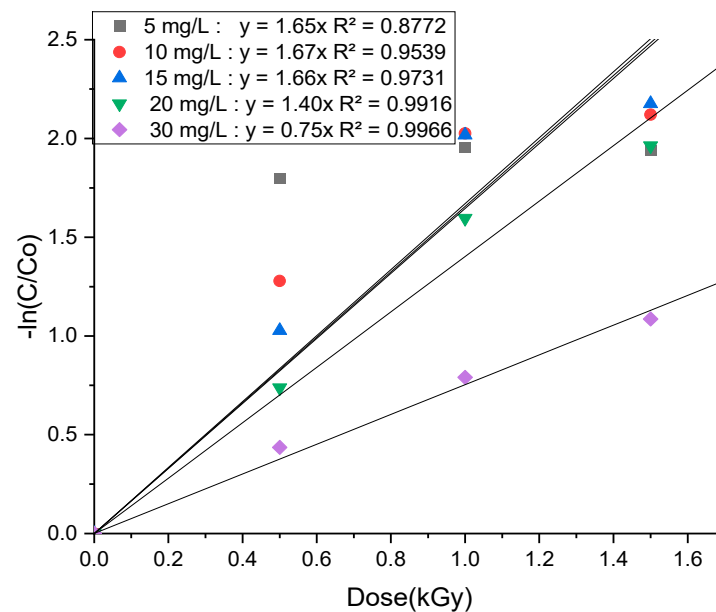
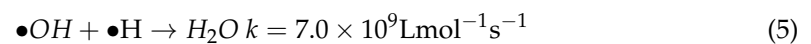
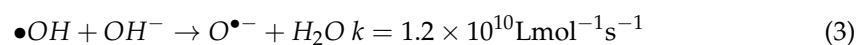


Figure 3. The kinetics of SDZ degradation.

3.3. Effect of Initial pH

Recent studies on pharmaceutical compounds such as chloroquine and hydroxychloroquine degradation under electron beam demonstrated the pH dependence in the removal efficiency of antibiotics [29,30]. Degradation of 30 mg/L SDZ was performed at various pH values (3.0, 6.3, 9.0, and 11.0) to ensure a proper analysis in the acidic, neutral, and alkaline environment. The degradation of sulfonamides under EB highly depends on the generation of reactive species ($\bullet\text{OH}$, e_{aq}^- and $\bullet\text{H}$), which react at different pH, as shown in Equations (3)–(5).



At pH 3, the degradation efficiency is more pronounced, reaching 80.8% at 3.0 kGy, suggesting that acidic condition enhances the reactivity of hydroxy radicals and hydrogen radicals. In acidic media, hydrated electrons are converted to hydrogen radicals as in Equation (4), thereby increasing the concentration of reactive species available to attack SDZ [31]. In contrast, at pH 6.3, the maximum degradation efficiency at 3.0 kGy reached 75.3% which is lower than at pH 3.0 but still significant. This gradual decrease may be due to the fact that proton availability is reduced, limiting the conversion of hydrated electrons to hydrogen radicals. An unfavorable degradation rate was noticed in alkaline medium that is 69.5% and 69.8% at pH 9.0 and 11.0, respectively. In the alkaline environment, the formation of hydroxyl radical is inhibited as in Equation (3) while hydrated electrons, which are less effective due to the lack of strongly electron-deficient sites in SDZ, dominate

the reaction environment. The degradation rate constants were 1.1, 0.9, 0.7, and 0.6 at pH 3.0, 6.3, 9.0, and 11.0, respectively. Therefore, as shown in Figure 4b, the degradation rate of SDZ at various pH conditions followed this order: pH 3.0 > 6.3 > 9.0 > 11.0.

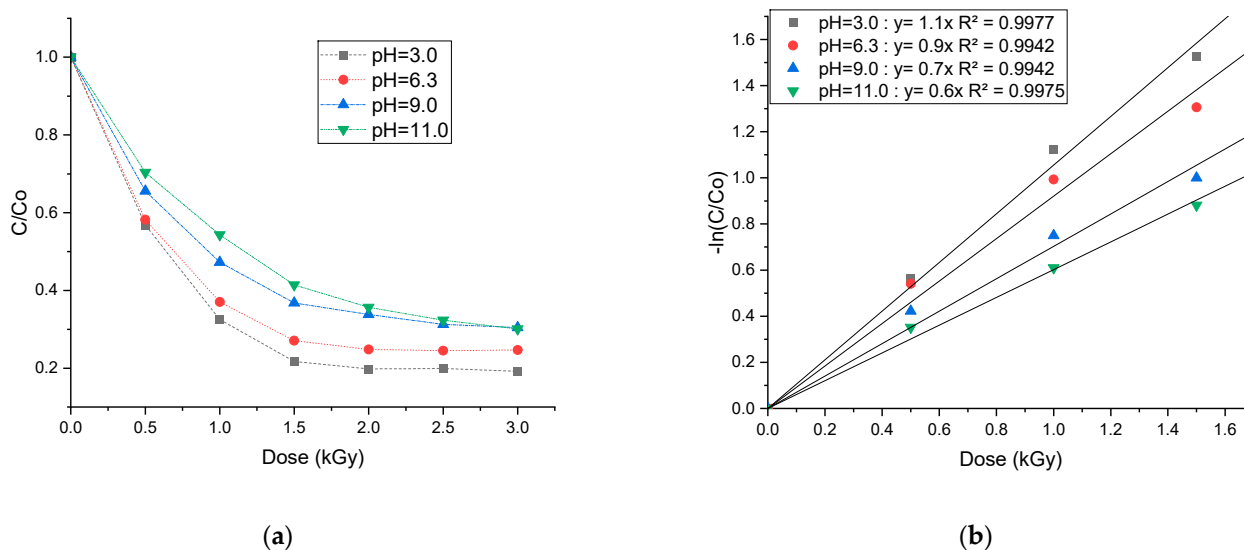


Figure 4. (a) Effect of the pH on the degradation of 30 mg/L SDZ (dose: 0–3 kGy). (b) Kinetic analysis.

3.4. Effect of Reactive Species on SDZ Degradation

The radiolysis of water is accompanied by the in situ generation of reactive species as in Equation (1). The three main species responsible for degradation of organic pollutants either by oxidation or reduction reactions are hydroxyl radicals ($\bullet OH$); hydrated electrons (e_{aq}^-); and hydrogen radicals ($\bullet H$) [32]. In order to evaluate the effect of hydroxyl radicals on the degradation of SDZ, the reaction was carried in an N_2O gas saturated system to scavenge hydrated electrons as in Equation (6).



To study the contribution of e_{aq}^- , 0.5 M tert-butanol in Ar gas saturated solution at pH 6.3 was used. In this system, tert-butanol could effectively scavenge $\bullet OH$ radicals following Equation (7).



To the aforementioned system for the evaluation of hydrated electrons, the pH of the solution was adjusted to less than 2 with 0.1M HCl in order to investigate the effect of hydrogen radicals ($\bullet H$) on the degradation of SDZ. With pH modification, hydrated electrons were converted to hydrogen radicals as shown in Equation (4) [33].

Figure 5a is a representative of the trend of the main reactive species on the degradation of SDZ under EB. The removal efficiency of SDZ followed the order $\bullet OH > \bullet H > e_{aq}^-$ due to the difference in reactivity of these species. Hydroxyl radicals (strong oxidant) play a significant role in the degradation of antibiotics due to their high reactivity and non-selective nature, leading to multiple site attacks on SDZ whereas hydrated electrons (strong reductant) are less reactive due to their selectivity and the specific electronic structures of target molecules. With regard to SDZ structure, the effect of hydrated electrons may be less pronounced due to the fact that SDZ lacks strong electron-deficient groups [33]. Under acidic conditions, more hydrogen radicals, which are moderately selective, interact with SDZ, enhancing the degradation of SDZ compared to neutral conditions.

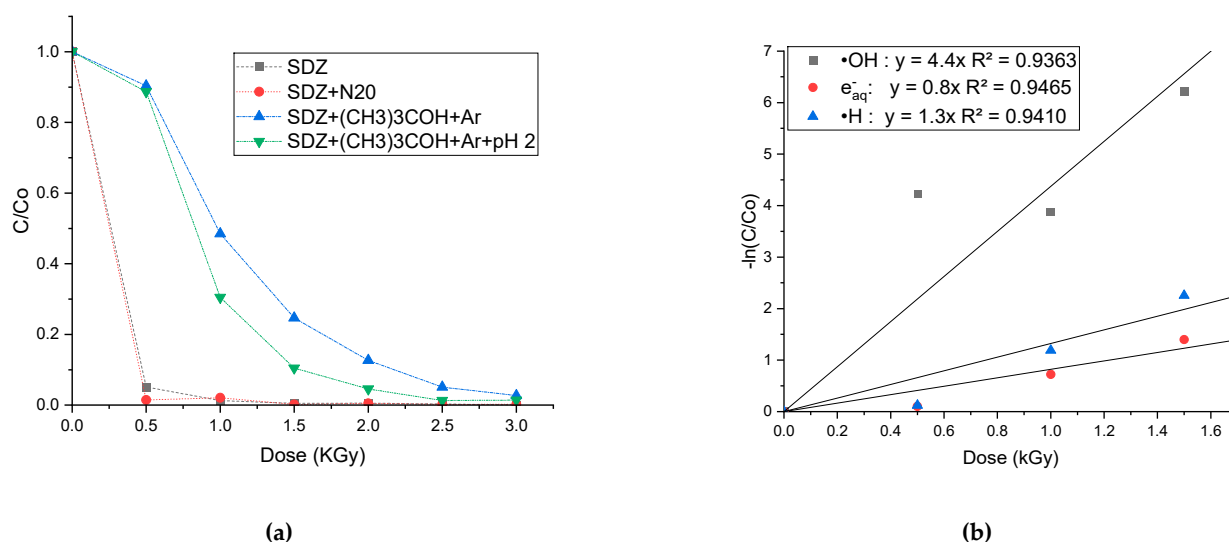


Figure 5. (a) Effect of reactive species on 10 mg/L SDZ degradation under EB irradiation monitored by HPLC (N₂O sparging = 30 min; Ar sparging = 30 min; 0.5 M tert-butanol; dose: 0–3 kGy); (b) kinetic analysis.

The degradation of SDZ followed a pseudo-first order kinetic with rate constants k obtained from the slopes of $-\ln\left(\frac{C}{C_0}\right)$ versus dose (kGy) Figure 5b. In the presence of N₂O, SDZ exhibited the highest degradation rate, with $k = 4.4 \text{ kGy}^{-1}$ ($R^2 = 0.9363$). This significant enhancement is attributed to the ability of N₂O to effectively convert solvated electrons into hydroxyl radicals ($\bullet OH$), increasing the oxidative degradation of SDZ. Conversely, when tert-butanol was added under an argon (Ar) atmosphere, the degradation rate was markedly reduced ($k = 0.8 \text{ kGy}^{-1}$, $R^2 = 0.9465$). Since tert-butanol is a well-known scavenger of hydroxyl radicals, this suggests that SDZ degradation is primarily driven by ($\bullet OH$), and its suppression significantly limits the breakdown process. At pH 2, in the presence of tert-butanol and Ar, an intermediate degradation rate was observed ($k = 1.3 \text{ kGy}^{-1}$, $R^2 = 0.9410$). The slight increase compared to pH 6.3 conditions suggests that acidic environments may enhance degradation, possibly by increasing the protonation of SDZ, which can influence its reactivity with residual radicals.

3.5. LC-MS Results

The identification of SDZ was achieved at 0 kGy with the help of LC-MS. Table 1. lists the major byproducts in solution after degradation of 10 mg/L SDZ under electron beam at 0.5 kGy and 1.0 kGy. Based on these intermediates, a degradation pathway could be proposed. The results suggest an interaction between $\bullet OH$ radicals and SDZ on N-H bonds at the benzene ring and hydroxylation at the pyrimidine ring of SDZ, which led to intermediate B. Further hydroxylation of B caused the formation of E, F, and G and the cleavage of the pyrimidine ring at the unstable C-N bonds to form intermediate H [34]. The identification of D indicated the cleavage of C-N bonds of the benzene ring; further hydroxylation led to the splitting of the sulfonamide functional group to J and I [3]. Intermediate C could refer to the result of the loss of sulfur dioxide from SDZ through oxidation [35]. Figure 6 provides insight into a plausible degradation mechanism of SDZ during EB irradiation primarily driven by hydroxyl radicals ($\bullet OH$) oxidation and the direct decomposition of SDZ molecules.

Table 1. The main intermediates during SDZ electron beam irradiation.

Compounds	Retention Time	Main Fragments (<i>m/z</i>)	Molecular Structure
A 4-amino- <i>N</i> -pyrimidin-2-yl-benzenesulfonamide (SDZ)	7.17	251	
B	15.74	283	
C	1.003	187	
D	1.003	236	
E	14.408	191	
F	1.003	145	
G	9.73	128	
H	18.494	229	
I	0.471	97	
J	0.471	141	

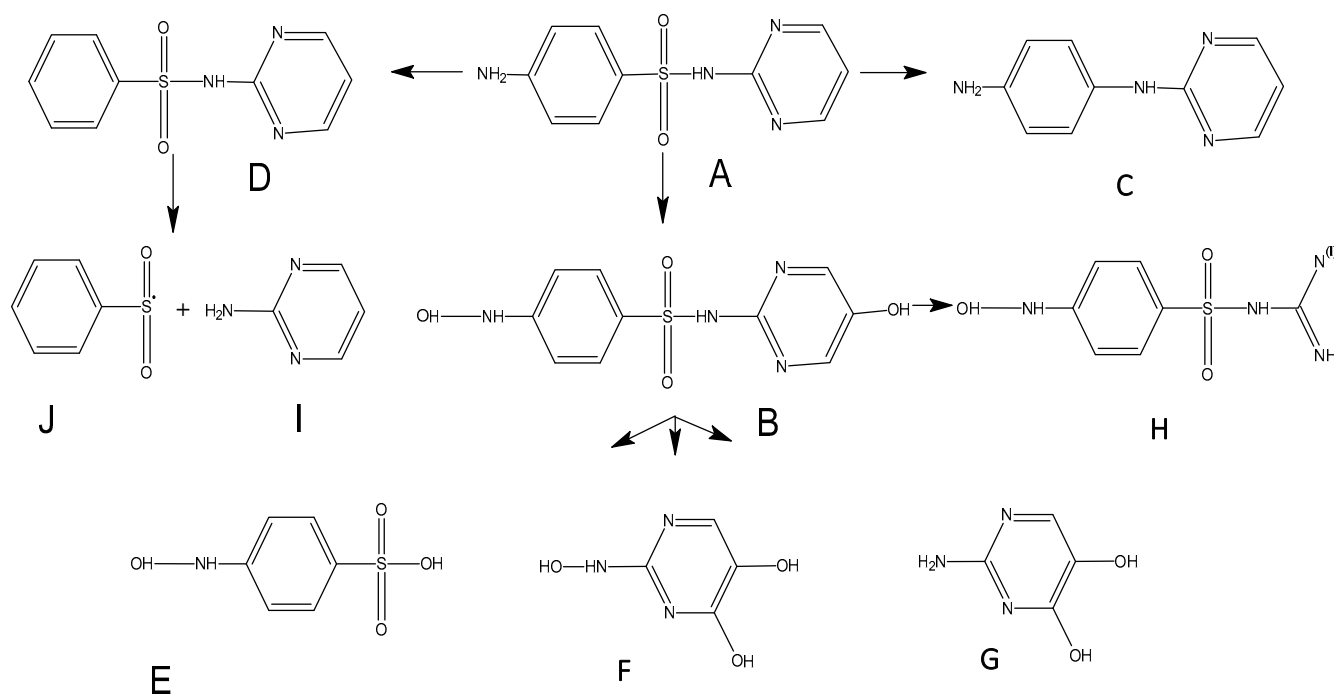


Figure 6. Proposed degradation pathway of SDZ by EBI (Identified intermediates: A–J).

4. Conclusions

This study investigated the degradation of sulfadiazine (SDZ) under electron beam (EB) irradiation, highlighting its dose-dependent breakdown, the influence of initial pH conditions, and the dominant role of reactive species in the degradation process. The UV–Vis spectral analysis confirmed a significant reduction in SDZ absorbance with increasing irradiation dose, with nearly complete degradation achieved at 3.0 kGy. The reaction kinetics followed a pseudo-first-order model, with degradation efficiency decreasing as SDZ concentration increased, likely due to radical competition between parent compounds and their byproducts.

The role of reactive species on SDZ degradation was examined, demonstrating that hydroxyl radicals ($\bullet OH$) were the most dominant oxidative agents, while hydrated electrons (e_{aq}^-) and hydrogen radicals ($\bullet H$) played secondary roles, influenced by pH conditions. Degradation efficiency was the highest under acidic conditions due to enhanced radical interactions. The identification of intermediate products through LC–MS analysis provided insight into plausible degradation pathways, primarily involving hydroxylation, oxidative cleavage, and desulfonation. This research confirms that EB irradiation is an effective method for SDZ degradation, offering a promising alternative for antibiotic removal in wastewater treatment.

Future research should focus on scaling up this process for real wastewater applications, evaluating its efficiency in complex matrices containing natural organic matter, salts, and competing pollutants. Further investigations into the toxicity of degradation byproducts are necessary to ensure the environmental safety of treated water. Additionally, combining EB irradiation with other Advanced Oxidation Processes (e.g., UV, ozonation, Fenton) could be explored to enhance degradation efficiency and reduce energy consumption. A techno-economic analysis would also be valuable in assessing the feasibility of EB irradiation as a large-scale water treatment solution.

Author Contributions: Conceptualization, Methodology, Supervision: Y.S.; Investigation: B.T.K., S.B., and S.W.; Data curation and formal analysis: B.T.K. and S.W.; Writing—original draft preparation:

B.T.K.; Writing—review and editing: Y.S. and J.W. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data presented in this study are available on request.

Conflicts of 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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