Comparison of EB-radiolysis and UV/H2O2-degradation of CBZ in pure water and solutions (Postprint)

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Abstract

Electron beam (EB) irradiation at 10 kGy achieved a 99% degradation rate for carbamazepine (CBZ) (75 mg L^{-1}) in solution, while UV and UV/H_2O_2 processes yielded degradation rates of 15.74% and 96.02%, respectively, for CBZ (2 mg L^{-1}) within 180 minutes. Based on energy consumption comparisons between EB irradiation and UV/H_2O_2, EB radiolysis was considered an efficient and energy-saving method for CBZ decomposition. Alkali metal cations present in surface water exhibited minimal inhibition of CBZ degradation under both EB and UV/H_2O_2 processes. Common anions in surface water showed little suppression of CBZ EB radiolysis, while inhibiting UV photodegradation of CBZ. Fe^{3+} enhanced degradation under both EB and UV irradiation due to its role as an $\bullet$OH promoter. Additionally, Fe^{2+} substantially enhanced CBZ degradation under UV irradiation. Based on the identification of intermediates during radiolysis, the primary degradation pathway for CBZ mineralization under EB irradiation was proposed. It was concluded that EB radiolysis is an efficient and energy-saving method for the complete mineralization of CBZ.

Full Text

Preamble

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Comparison of EB-radiolysis and UV/H_2O_2 Degradation of CBZ in Pure Water and Solutions

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Abstract: This study demonstrates that a 99% degradation rate of carbamazepine (CBZ) (75 mg/L) in aqueous solutions can be achieved under 10 kGy of electron beam (EB) irradiation, while UV and UV/H_2O_2 processes achieve only 15.74% and 96.02% degradation of CBZ (2 mg/L) in 180 minutes, respectively. EB-radiolysis is identified as an efficient and energy-saving method for CBZ decomposition compared to UV/H_2O_2 when comparing energy consumption. Alkali metal cations commonly present in surface water exhibit minimal inhibition on CBZ degradation under both EB and UV/H_2O_2 processes. Anions typically found in surface water show little suppression on EB-radiolysis of CBZ but cause significant inhibition of UV-photodegradation. Fe^{3+} promotes CBZ degradation under both EB irradiation and UV irradiation by acting as an $OH\cdot$ promoter, while Fe^{2+} substantially enhances CBZ degradation under UV irradiation. Based on the intermediates identified during radiolysis, a main degradation pathway for CBZ mineralization is proposed under EB irradiation. It is concluded that EB-radiolysis is an efficient and energy-saving method for complete CBZ mineralization.

Keywords: Carbamazepine, Electron beam radiolysis, UV-photodegradation, Ions, Intermediates
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Introduction

Carbamazepine (CBZ) is a widely consumed psychotropic pharmaceutical and one of the most frequently detected persistent pharmaceuticals in the environment [1]. It contaminates both surface water and groundwater [2], with concentrations reaching up to 6.3 µg/L in wastewater, 1.1 µg/L in surface water, and 30 ng/L in drinking water [3]. CBZ is refractory to both conventional and advanced wastewater treatment processes [4, 5] and can significantly affect aquatic organisms [5].

Transformation of carbamazepine by various means has been reported, including biotechnology [5–10] and advanced oxidation processes (AOPs). However, traditional oxidation processes do not always achieve complete mineralization. Hu reported only 7% removal efficiency of CBZ by conventional wastewater treatment [11], and Tixier observed only slight elimination through phototransformation [12].

Electron beam (EB) treatment is an advanced oxidation process [13] that is efficient, safe, simple, and capable of complete mineralization of organic pollutants, especially for thorough decomposition of biorefractory compounds [14–19]. In EB-radiolysis, water decomposes into hydroxyl radicals ($OH\cdot$), hydrogen radicals (H•), and hydrated electrons (e^-aq) with different G-values (µmol/J), with OH• and e^-aq being the main active species [20].

This paper compares the degradation efficiency and energy consumption of EB-radiolysis and UV/H_2O_2 degradation of CBZ in pure water. Since various ions are present in surface water, their effects on CBZ degradation by EB and UV/H_2O_2 treatments were investigated. The transformation pathway of CBZ under EB irradiation is proposed. These findings will help identify optimal methods for treating pharmaceutical and personal care products (PPCPs) in surface water.

II. Materials and Methods

A. Materials

CBZ (>98%), methanol (HPLC grade), and acetonitrile (LC-MS grade) were obtained from Sigma-Aldrich. Formic acid (HCOOH), acetic acid (CH_3COOH), oxalic acid ($H_2C_2O_4$), NaCl, Na_2SO_4, $NaHCO_3$, $CaCl_2$, $MgCl_2$, $FeSO_4$, $Fe_2(SO_4)_3$, $FeCl_2$, and $FeCl_3$ were purchased from Shanghai Chemical Reagent Co., Ltd. All chemicals were of analytical grade unless otherwise stated. Pure water (resistance >18.2 MΩ) was prepared using a Millipore Milli-Q system. Surface water was collected from a local river and filtered through 0.22 µm filters for degradation experiments. All experiments were performed at 20 ± 2 °C.

B. Photo Irradiation Procedure

Photodegradation experiments were conducted using a merry-go-round photochemical reactor (Sidongke Electric Plant, Nanjing, China) equipped with a maximum of 12 quartz tubes containing the reaction solution. Each tube had a total volume of 100 mL and a liquid layer thickness of 35 mm. UV irradiation was provided by a 300 W high-pressure mercury lamp with 254 nm wavelength, immersed in a circulated-water cooled quartz well. The light intensity was 9 W and the cell path length was 2 cm. The reactor temperature was maintained at 20 ± 2 °C by a water-cooling jacket.

C. EB Irradiation

Samples were irradiated at ambient temperature with 1.8 MeV electron beams at currents up to 10 mA from an electron accelerator, receiving doses of 0.5, 1, 2, 5, 10, and 20 kGy. Samples were placed 30 cm below the beam scanner.

D. Analytical Methods

CBZ Concentration Analysis: CBZ concentration was measured by high-performance liquid chromatography (HPLC, Agilent 1200 series) using a C18 column (150 mm × 4.6 mm) with an autosampler injecting 10 µL volumes.

Detection was performed at 230 nm using a VWD detector. The mobile phase consisted of methanol and water (55:45, v:v) at a flow rate of 1.0 mL/min.

Ion Analysis: Organic acids, nitrate, nitrite, and ammonium ions produced by EB-radiolysis were detected using an ICS1100 ion chromatography system (Dionex). An anion exchange column (IonPac AS22, analytical, 4 mm × 250 mm) was used with an eluent mixture of 4.5 mM Na_2CO_3 and 1.4 mM $NaHCO_3$ at 1.20 mL/min flow rate, with 25 µL injection volume. The suppressor was an Anion Self-Regenerating Suppressor (ASRS 300 4 mm) under AutoSuppression Recycle Mode at 31 mA. For cations, a hydrophilic cation exchange column (IonPac CS12A, analytical, 4 mm × 250 mm) was used with 20 mM methanesulfonic acid eluent at 1 mL/min flow rate and 25 µL injection volume. The suppressor was a Cation Self-Regenerating Suppressor (CSRS ULTRA II, 4 mm) under Auto-Suppression Recycle Mode at 59 mA.

By-product Analysis: CBZ degradation by-products were monitored by LC/MS/MS using an Agilent 1260 LC chromatograph coupled to an Agilent 6460 mass spectrometer with electrospray ionization (ESI) interface and a heated nebulizer. Separation was performed on a Porshell 120 100 mm × 3 mm EC-C18 end-capped column (2.7 µm particle size) at 0.4 mL/min flow rate with 10 µL injection volume. The mobile phase consisted of acetonitrile (A) and 0.1% HCOOH in water (B) with a gradient from 5% to 95% A over 8 min, held at 95% A for 2 min, then at 100% A for 2 min, and returned to initial conditions in 3.5 min. Mass spectrometry full scanning analysis was performed from 50-500 m/z. For positive ESI (+), source conditions were: capillary voltage 4000 V, nebulizer pressure 40 psi (0.28 MPa), drying gas flow 8 L/min at 300 °C, and nozzle voltage 0 V. For negative ESI (-), conditions were: capillary voltage 3250 V, nebulizer pressure 40 psi, drying gas flow 7 L/min at 350 °C, and nozzle voltage 500 V.

TOC Analysis: Total organic carbon was determined using a TOC analyzer (multi C/N 2100, Analytik Jena AG).

III. Results and Discussion

A. UV-Photodegradation and EB-radiolysis of CBZ in Pure Water

As shown in [Figure 1: see original paper], only 15.74% of CBZ (2 mg/L) was degraded after 180 minutes of UV radiation alone, while 96% degradation was achieved under UV/H_2O_2, and 99.9% degradation of CBZ (75 mg/L) occurred under 10 kGy EB irradiation. These results demonstrate that CBZ is refractory to UV irradiation alone, while H_2O_2 addition significantly promotes degradation. EB-radiolysis proves to be an efficient method for CBZ degradation, consistent with reports by Kwon et al. [16].

The EB-radiolysis enhancement is attributed to the large quantities of hydroxyl radicals (OH•), hydrogen radicals (H•), and hydrated electrons (e^-aq) generated during EB irradiation [20] at different G-values (µmol/J) (Reaction I).

As is well known, OH•, e^-aq, and H• are highly reactive species that degrade organic compounds. The OH•radical is a powerful oxidant with an oxidation potential (E_0 = 2.8 V) that can oxidize organic compounds in aqueous solutions, while e^-aq and H• are strong reductants. The hydrated electron possesses a particularly strong reducing ability (E_0 = -2.9 V) that effectively dehalogenates halogenated organic compounds [14].

The photooxidation of organic substances follows the line source spherical emission model proposed in previous studies [22]. Similar to the above approach, when 90% of CBZ is degraded, the energy consumption Q_2 (J/mol) can be calculated using Equation (1):

$$Q_2 = (T_{0\cdot 9}I(0,\lambda)M)/(\Delta CL)$$

where I_0,λ (Einstein/(L•s)) is the intensity of incident light at 253.7 nm (9 W), $T_{0\cdot 9}$ is the time required for 90% CBZ degradation, M is the molecular mass of carbamazepine (236.27 g/mol), ΔC is the concentration change at 90% degradation, and L is the CBZ solution volume (1.350 L).

The low degradation percentage of CBZ under UV treatment alone suggests limited direct photolysis. In contrast, the numerous oxidative free radicals produced in the UV/H_2O_2 process (Reactions II-IV) react effectively with organic pollutants [21]. The results indicate that CBZ removal in the UV/H_2O_2 system is likely controlled by the availability of hydroxyl radicals. Therefore, both EB irradiation and UV/H_2O_2 are efficient methods for decomposing refractory organic pollutants, with EB irradiation being simpler and more convenient as it requires no added oxidizing agents.

Reaction I: $H_2O \rightarrow (0.28)OH\bullet + (0.27)e^-aq + (0.06)H\bullet + (0.05)H_2 + (0.07)H_2O_2 + (0.27)H^+$

Reaction II: $H_2O_2 + h \rightarrow 2OH\bullet$

Reaction III: $OH\bullet + OH\bullet \rightarrow H_2O_2$

Reaction IV: $H_2O_2 + OH\bullet \rightarrow HO_2\bullet + H_2O$

B. Comparison of EB-radiolysis and UV/H_2O_2 Photodegradation Efficiency

To compare energy consumption between EB radiation and UV/H_2O_2 treatments, CBZ initial concentrations of 20 mg/L and 25 mg/L were used at pH 6.3. Degradation kinetics for both treatments follow pseudo-first-order kinetics described by $\ln(C/C_0) = -kx$, where C and C_0 are residual and initial CBZ concentrations (mg/L), k is the pseudo-first-order rate constant, and x is either absorbed dose (kGy) for EB irradiation or time (min) for UV/H_2O_2 process. Table 1 presents the fitted rate constants for different initial concentrations.

In EB degradation, $D_{0\cdot 90}$ (kGy) represents the absorbed dose required for 90% degradation efficiency. We defined Q_1 (J/mol) as the energy consumption for

90% CBZ degradation, calculated using Equation (3):

$Q_1 = D_{0 \cdot 90}M$

where $D_{0 \cdot 90}$ (kGy) is the dose at which 90% CBZ is degraded and M is the molecular mass of carbamazepine (236.27 g/mol).

As shown in , the EB process is more efficient than UV/H_2O_2, with the latter showing higher Q values. A smaller Q value indicates better efficiency, confirming that EB irradiation is an efficient method for CBZ degradation.

C. Effect of Ions on CBZ Degradation

1. Effect of Salts on CBZ EB-radiolysis CBZ (75 mg/L) solutions containing different cations (NaCl, $CaCl_2$, $MgCl_2$, and $FeCl_3$, 5 mM each) or anions (NaCl, Na_2SO_4, and $NaHCO_3$, 5 mM each) were EB-irradiated to assess their effects. Experiments were performed in triplicate with coefficient of variation within 5%. As shown in [Figure 2: see original paper], Na^+, Mg^{2+}, and Ca^{2+} cations, as well as Cl^-, ${SO_4}^{2-}$, and ${HCO_3}^-$ anions, had minimal impact on EB-radiolysis of CBZ, while Fe^{3+} promoted degradation by acting as an $OH\cdot$ promoter.

2. Effect of Salts on CBZ Photodegradation CBZ (2 mg/L) solutions containing NaCl, $CaCl_2$, Na_2SO_4, or $NaHCO_3$ (10 mM each) were prepared with 20 mM H_2O_2 and irradiated under a 300 W mercury lamp. Samples were taken at intervals of 0, 5, 10, 20, 40, 60, 90, and 120 minutes. Experiments were performed in triplicate with variation within 2%.

As shown in [Figure 3: see original paper], $NaHCO_3$ showed greater inhibition (32.93% degradation) than Cl^- (78.60% degradation) at 120 minutes. This may occur because ${HCO_3}^-$ reacts with $OH\cdot$ to produce ${CO_3}\cdot^-$, which has lower oxidative activity than $OH\cdot$ [23, 24]. Similar to ${HCO_3}^-$, NaCl and $CaCl_2$ showed slight inhibition of CBZ degradation because Cl^- can also scavenge $OH\cdot$, competing with CBZ for $OH\cdot$ radicals [25].

3. Effect of Fe^{2+} and Fe^{3+} on CBZ Photodegradation Experiments were conducted by adding 2.0 mM $FeSO_4$ or 1.0 mM $Fe_2(SO_4)_3$ to CBZ solutions (2.0 mg/L, pH=3) and photodegrading under 300 W UV. Experiments were performed in triplicate with coefficient of variation within 5%.

As shown in [Figure 4: see original paper], CBZ removal efficiency increased significantly with Fe^{2+} and Fe^{3+} addition. Oxidation of Fe(II) to Fe(III) produces H_2O_2 and $OH\cdot$ in the presence of O_2 (Reactions V–VIII). Fe(III) ions generate hydroxyl radicals under UV irradiation (Reaction IX), which react with organic substances to form organic radicals (Reaction X).

Reaction V: $Fe^{2+} + O_2 \rightarrow Fe^{3+} + {O_2}\cdot^-$

Reaction VI: $Fe^{2+} + {O_2}\cdot^- + 2H^+ \rightarrow Fe^{3+} + H_2O_2$

Reaction VII: $Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH\cdot$

Reaction VIII: $Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2\cdot + H^+$

Reaction IX: $Fe^{3+} + H_2O + h \rightarrow Fe^{2+} + H^+ + OH\cdot$

Reaction X: $OH\cdot + RH \rightarrow H_2O + R\cdot$

Therefore, anions present in natural water significantly affect UV-degradation of CBZ but have minimal effect on EB-radiolysis. Alkali metal cations have little influence on both UV- and EB-degradation of CBZ, while Fe^{3+} enhances CBZ degradation. Thus, EB irradiation is an ideal method for degrading contaminants in solutions containing various salts.

D. Degradation Process of CBZ by EB Irradiation in Ionic Solutions

Intermediates of CBZ in solutions containing all ions were detected by IC and LC/MS/MS. Organic acids identified included formic, acetic, oxalic, malonic, and succinic acids. Inorganic ions detected were nitrite and nitrate (by anion IC) and ammonium (by cation IC). Nitrite and nitrate disappeared after 15 kGy, indicating that organic nitrogen was primarily transformed to ammonium ion (NH_4^+) or N_2. Other complex transformations are summarized in .

[Figure 5: see original paper] shows the effect of radiation dose on TOC removal efficiency and NH_4^+ concentration evolution for CBZ solutions. At 20 kGy, residual TOC was approximately 20%, and NH_4^+ concentration was about 2.01 mg/L. The calculated total organic nitrogen was 3.81 mg/L, suggesting that 52.8% of organic nitrogen was converted to ammonia nitrogen and about 25% was transformed to N_2.

Based on intermediate detection by LC/MS/MS and ion variation by IC during radiolysis, a main degradation pathway for CBZ mineralization is proposed in [Figure 6: see original paper]. A 20 kGy EB-irradiation achieved nearly complete mineralization of CBZ to CO_2, H_2O, and mineral nitrogen, whereas UV/H_2O_2 photodegradation required much longer time for CBZ mineralization.

IV. Conclusion

Comparison of EB-radiolysis and UV-photodegradation of CBZ in pure water reveals that CBZ is readily degraded under EB irradiation but refractory under UV irradiation alone. While the degradation rate is markedly enhanced under UV/H_2O_2 process where $OH\cdot$ plays a vital role, EB radiation proves more efficient when comparing energy consumption between EB irradiation and UV/H_2O_2 processes.

Ions have minimal influence on EB-radiolysis of CBZ but inhibit CBZ degradation under UV/H_2O_2 process. Fe^{2+} and Fe^{3+} substantially enhance CBZ UV-photodegradation. Therefore, EB irradiation is an efficient method for degrading contaminants in surface water containing various salts. Furthermore, intermediates detected by LC/MS/MS and IC show that CBZ can be completely mineralized into CO_2 (detected by TOC analyzer), H_2O, and mineral nitrogen. Electron beam technology is thus an efficient and energy-saving method for CBZ decomposition and shows promise for application in treating other persistent PPCPs.

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