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Electron beam treatment for the removal of 1,4-dioxane in water and wastewater

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ABSTRACT

Electron beam (e-beam) treatment uses accelerated electrons to form oxidizing and reducing radicals when applied to water without the use of external chemicals. In this study, electron beam treatment was used to degrade 1,4-dioxane in several water matrices. Removal improved in the progressively cleaner water matrices and removals as high as 94% to 99% were observed at a dose of 2.3 kGy in secondary effluent. 1,4-dioxane removal was confirmed to be primarily through hydroxyl radical oxidation. The calculated electrical energy per order was found to be 0.53, 0.26, and 0.08 kWh/m^3/order for secondary effluent (Avg. TOC 9.25 mg/L), granular activated carbon effluent (TOC 3.46 mg/L), and ultrapure water, respectively, with a 70% generation and transfer efficiency applied.

Key words: advanced oxidation, 1, 4-dioxane, electron beam, wastewater treatment, water treatment

HIGHLIGHTS

- Electron beam treatment was used to successfully remove 1,4-dioxane from water and wastewater.
- No bromate was formed despite high bromide concentrations and bromate removal was observed.
- Energy use was comparable to other conventional AOPs without requiring additional chemicals.
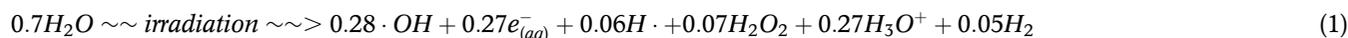
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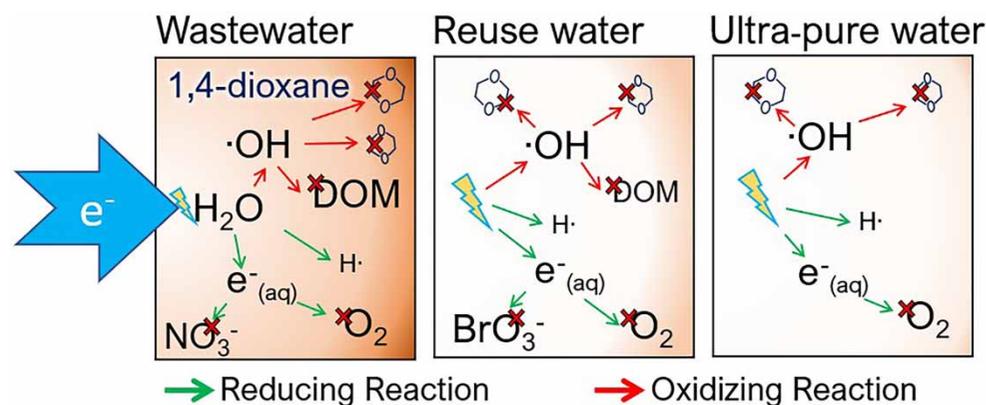
1. INTRODUCTION

1,4-dioxane is a prevalent trace contaminant in water and wastewater treatment and a potential human carcinogen with an EPA estimated 10^{-6} lifetime cancer risk associated with a concentration of 0.35 μg/L (USEPA 2013). It was historically used as a solvent stabilizer for 1,1,1-trichloroethane (TCE) and is a common groundwater and surface water contaminant and was detected in 21% of US drinking water supplies (Adamson et al. 2017). It is also commonly found in household detergents and

cleaning products which end up in wastewater treatment plant influents (Tanabe & Kawata 2008). Because it is not readily biodegraded, adsorbed, or oxidized by traditional oxidants, it can be very difficult to treat (Otto & Nagaraja 2007).

Advanced oxidation processes (AOPs) are typically used to remove 1,4-dioxane (Otto & Nagaraja 2007; McElroy et al. 2019). In these processes, powerful oxidizing hydroxyl radicals (·OH) are generated in-situ to degrade contaminants. Technologies include UV advanced oxidation (UVAOP) where UV light is used for photolysis of hydrogen peroxide (H_2O_2) into ·OH. Likewise, UV-Chlorine AOP generates ·OH from photolysis of free chlorine. Ozone (O_3) is applied as an oxidant and disinfectant in water and wastewater treatment and leads to the generation of ·OH through its autocatalytic decomposition and reactions with organics which can also be catalyzed by H_2O_2 (von Sonntag & von Gunten 2012; Stefan 2018). In these processes, large chemical doses are often needed. While the study of AOPs for 1,4-dioxane removal in wastewater effluent is limited, ozone is generally thought to be the best AOP for wastewater effluents. This is due to the high levels of dissolved organic matter which scavenges hydroxyl radicals and low transmittance of UV light (Katsoyiannis et al. 2011; Lee et al. 2016; Sgroi et al. 2021). However, ozone (and UV-Cl_2) can form bromate as a regulated disinfection byproduct if bromide (Br^-) is present.

Electron beam treatment uses accelerated electrons to generate large quantities of both oxidizing and reducing radicals through the radiolysis of water as shown in Equation (1) (Cooper et al. 1992; Wang et al. 2016):

$$0.7H_2O \sim\sim irradiation \sim\sim> 0.28 \cdot OH + 0.27e_{(aq)}^- + 0.06H \cdot + 0.07H_2O_2 + 0.27H_3O^+ + 0.05H_2 \tag{1}$$

This combination of oxidation and reduction is a major benefit over traditional advanced oxidation processes. The process has been used to treat pharmaceuticals such as carbamazepine and diclofenac in water (Tominaga et al. 2018; Zheng et al. 2020), industrial wastewaters (Han et al. 2002) and sludge (Farooq et al. 1993; Kim et al. 2020). Aqueous or hydrated electrons, $e_{(aq)}^-$, can also reduce bromate. Electricity is the only major input and no additional chemicals are required to generate the oxidizing and reducing species (Ponomarev & Ershov 2020). 60% to 80% electrical conversion and transfer efficiency is typical (Stefan 2018). Recently, the first full-scale application of e-beam treatment was applied at 30,000 m^3/day for aniline and color removal from an industrial wastewater (Wang et al. 2022). Doses are typically given in kilogray (kGy), where 1 kGy equals 1 kJ absorbed/kg or approximately 0.278 kWh/m^3 in water.

The objectives of this study were to (1) evaluate the performance of electron beam treatment for 1,4-dioxane removal in several real water matrices used for water and wastewater treatment and indirect potable reuse with high bromide, (2)
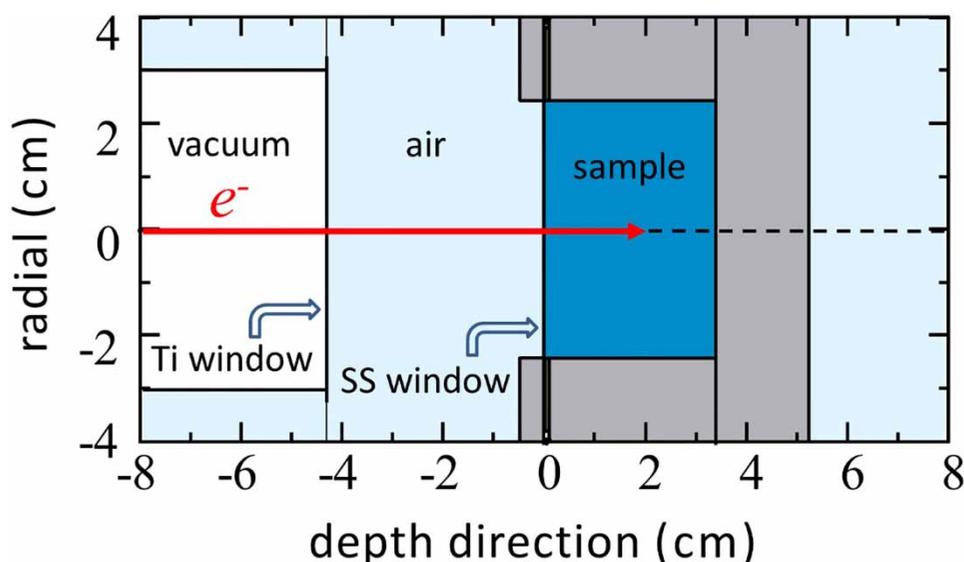
Figure 1 | Schematic layout of the water irradiation target. The electrons travel from left to right, out of the beamline vacuum, through a Ti-foil window, air, and a stainless steel window into the water sample.

determine the mechanisms of 1,4-dioxane degradation and the effects on other water quality parameters of interest, and (3) determine the electrical energy per order (EEO) for 1,4-dioxane removal by e-beam treatment.

2. MATERIALS AND METHODS

The Upgraded Injector Test Facility (UITF) is a superconducting radio-frequency accelerator at the Thomas Jefferson National Accelerator Facility (TJNAF i.e. JLab) capable of accelerating an electron beam up to an energy 10 MeV and a current of 100 nA, in continuous-wave mode. A dedicated beamline was added to investigate electron beam treatment on water samples. In this beamline, the water targets serve as a termination of the electron beam (Li *et al.* 2022). The sample target containers were constructed from solid aluminum blocks with a cylindrical hole to hold a sample volume of 60 mL. Figure 1 shows a schematic layout of the end of the beamline and the target sample holder. Four containers were placed on the target rail at the end of the irradiation beamline, the central position was taken by an X-ray screen on a dummy target (Figure S1). Each target was then moved in front of the beam remotely.

For the irradiation studies, the UITF was operated at an electron beam energy of 8 MeV and an average beam current of 108 nA, yielding an average beam power of 808 mW. The uncertainty in the beam power is ∼1%. During commissioning of the setup, the irradiation dose rate was calibrated by the use of dedicated dosimeter rods placed inside and in front of the sample holders, as well as corresponding simulation studies. During the water irradiation, the dose was varied from 0.25 kGy to 20 kGy for the individual samples by varying the beam exposure time from 30 seconds to 40 minutes. A detailed description of the beamline, the relevant electromagnetic elements, dosimetry measurements and simulations can be found in Li *et al.* (2022).

2.2. Sample water collection and analysis

Secondary effluent was collected from the Hampton Roads Sanitation District Nansemond Treatment Plant (Suffolk, VA). The plant consists of a 5-stage bardenpho process with methanol addition for denitrification. For a sample with lower total organic carbon and further treatment representing drinking water, granular activated carbon (GAC) effluent was collected from the SWIFT Research Center at the Nansemond Treatment Plant. In this process secondary effluent is treated through coagulation-flocculation and sedimentation, ozonation and biofiltration, followed by GAC adsorption and UV disinfection for indirect potable reuse via managed aquifer recharge. The design and operation of this plant has been described elsewhere (Gonzalez *et al.* 2021; Hogard *et al.* 2021). Ultra-pure water (UPW), resistivity >16 MΩ, was produced by a Smart2Pure UV Ultrapure Water System (Thermo-Fisher Scientific, Waltham, MA).

Secondary effluent samples were collected on three separate occasions, filtered, spiked with 1,4-dioxane (when applicable), refrigerated, and taken in a cooler to Jefferson Lab where they were refrigerated until use. Samples were then allowed to come up to room temperature, 22 °C, before irradiation. The secondary effluent and GAC effluent samples were filtered through a 0.45 µm nylon filter in order to remove turbidity and pathogens and limit the potential for biodegradation of the water matrix constituents during storage. TOC ranged from 8.98 to 10.74 mg/L in the secondary effluent, pH ranged from 7.0 to 7.2. pH was not measured at the time of testing; however, it is unlikely to have changed significantly over the course of sample storage. GAC samples were collected on two separate days, TOC was 3.47 and 3.45 mg/L, respectively. pH was 6.82 in the second sample at the time of sample collection and preparation

For most tests, a stock solution of reagent grade 1,4-dioxane diluted in ultra-pure water was spiked into the sample water to allow high enough concentrations for extraction and detection. For the test with the background level of 1,4-dioxane, two samples were irradiated at the desired dose and then combined into one 125 mL bottle. 1,4-dioxane samples were collected in 40 mL or 125 mL bottles preserved with 1 g/L sodium bisulfate depending on the desired detection limit. Samples were extracted by solid phase extraction and analyzed by a modified version of EPA method 522 by HRSD's Central Environmental Laboratory. Due to the limited sample volumes irradiated, replicate samples were not taken. Duplicate initial samples were taken on two occasions and measured within 6.5% difference (Table S1).

UV absorbance was measured on a Genesys UV-Vis spectrophotometer (Thermo-Fisher Scientific, Waltham MA). Total organic carbon and total nitrogen were measured on a Shimadzu TOC 4200 analyzer. Chemical oxygen demand (COD), nitrite and nitrate were measured by TNT methods 821, 839, and 835, respectively, with a Hach DR3900 Spectrophotometer. Dissolved oxygen (DO) was measured using a Hach LDO handheld DO probe (Hach Co, Loveland CO). Bromate was analyzed by ion chromatography by EPA method 300.1. Bromide was measured by EPA method 300.
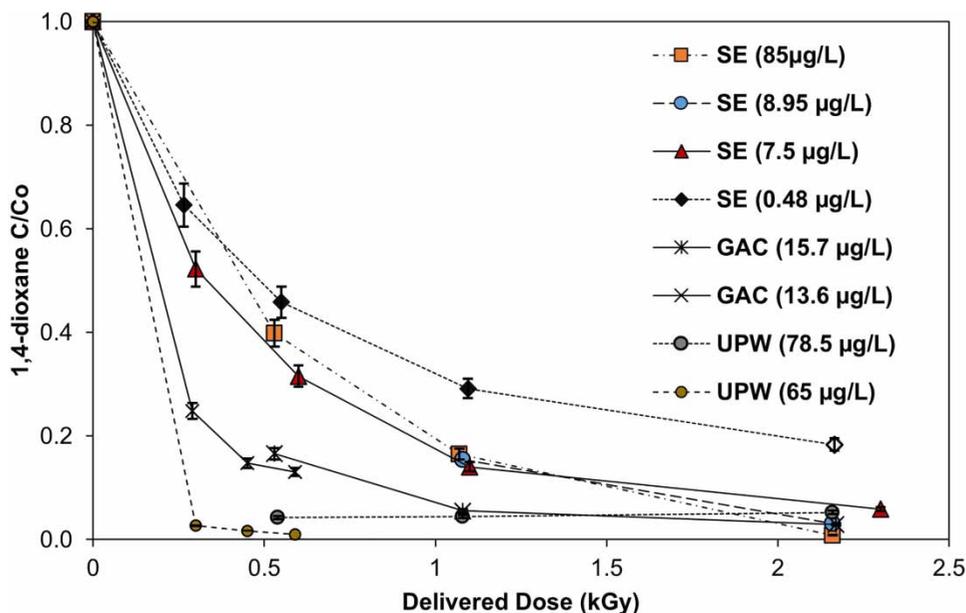
Figure 2 | 1,4-dioxane concentration vs delivered dose in SE, Secondary effluent, GAC, GAC effluent, UPW, ultrapure water. Initial concentrations in parentheses. The open diamond represents a value below the method reporting limit. Error bars represent the expected analytical error of $\pm 6.5\%$ applied to the final concentration.

3. RESULTS AND DISCUSSION

3.1. Removal of 1,4-dioxane

Samples of secondary effluent, GAC effluent, and ultrapure water spiked with 1,4-dioxane were irradiated at varying doses as shown in Figure 2. The reported doses represent the average dose or total energy absorbed by the sample. The relative uncertainty in the dose measurement was in the range 3%–5% for all samples. Removal improved with increasing e-beam dose and it was greater in the progressively cleaner water matrices. For the secondary effluent samples, lesser removal was observed for the samples with the background concentration of 1,4-dioxane (0.48 μg/L) while the removal was quite similar between the
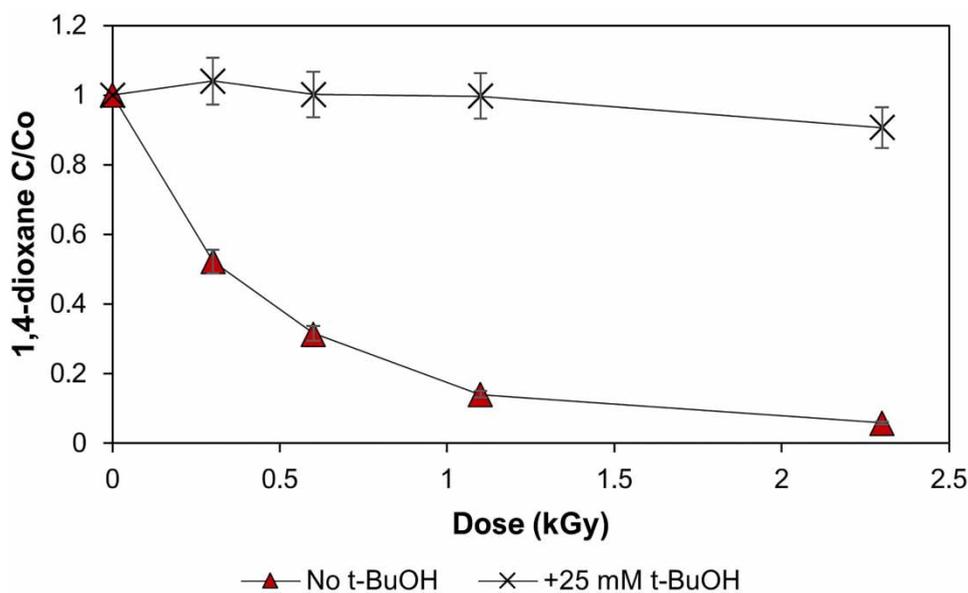
Figure 3 | 1,4-dioxane removal with and without tert-butanol. 7.5 μg/L initial 1,4-dioxane. Error bars represent the expected analytical error of $\pm 6.5\%$ applied to the final concentration.
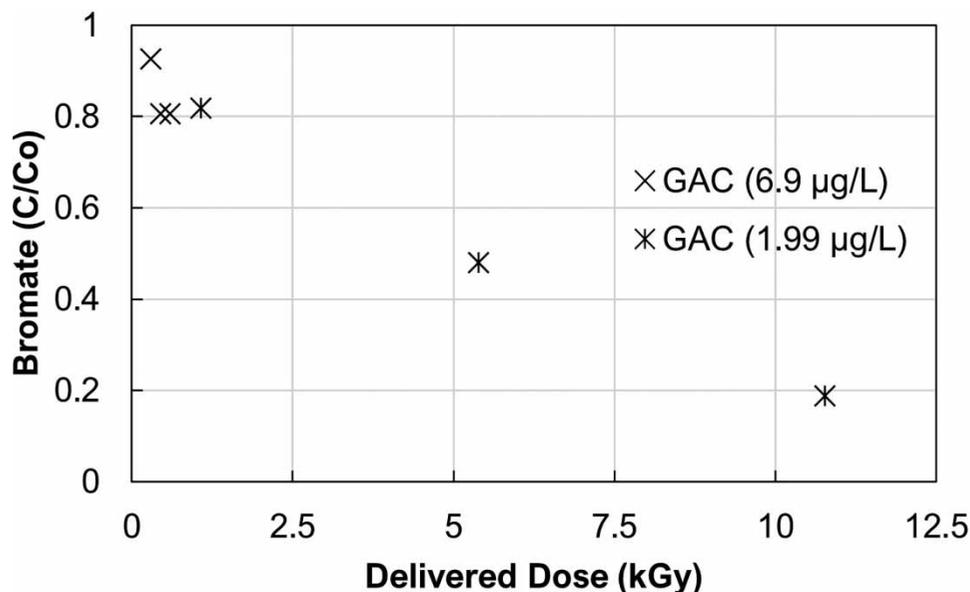
Figure 4 | Bromate removal in GAC effluent samples. Initial concentrations in parentheses.
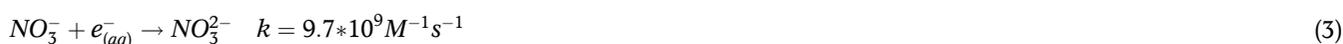
spiked samples at high and low concentrations. Because of the low concentration, the background samples had the greatest possibility for error, as they required two irradiation runs to generate sufficient sample volume for extraction and analysis.
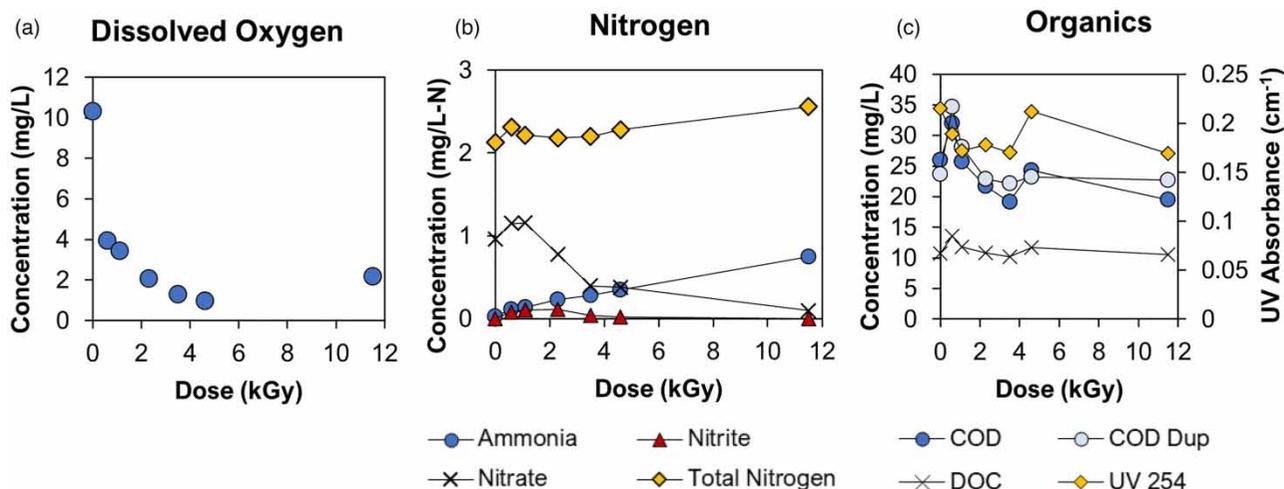
In some cases, moderate increases in dose did not continue to remove 1,4-dioxane as shown in the UPW results in Figure 2, though it could be decreased to below the method reporting limit (MRL) with doses >5 kGy (Table S1). There was a non-linear relationship between log-removal and dose (Figure S2) which could be due to the non-uniform distribution of dose through the sample due to the shallow penetration depth of the accelerated electrons and narrow spread of the beam over the sample. Dosimetry measurements and simulated dose distributions showed that the dose was concentrated toward the center of the sample holders and that ~10% of the sample volume at the back of the sample holders received less than 15% of the peak dose (Li *et al.* 2022). On the other hand, once the major $e_{(aq)}^-$ scavengers are consumed, $e_{(aq)}^-$ may react with ·OH and decrease 1,4-dioxane removal, though this seems less likely.

In one test shown in Figure 3, 25 mM tert-butanol (t-BuOH) was added as a hydroxyl radical scavenger and resulted in no removal of 1,4-dioxane, except for at the highest dose of 2.3 kGy where ~10% reduction occurred. Without t-BuOH addition at the same dose, 94% removal of 1,4-dioxane was observed. At the highest doses, hydrogen atom reduction may play a small part in the degradation of 1,4-dioxane. However, with a second order rate constant of $1*10^7$ $M^{-1}s^{-1}$ (Buxton *et al.* 1988), which is two orders of magnitude slower than ·OH ($k·_{OH} = 3.0*10^9$ $M^{-1}s^{-1}$), along with the lower yields of H· and H· scavenging by dissolved oxygen, it is unlikely to play a significant role. 25 mM t-BuOH is a concentration typically used in ozonation experiments at low doses (Buffle *et al.* 2006). However, others have used concentrations as high as 0.5 M to completely scavenge ·OH in irradiation experiments (Trojanowicz *et al.* 2021). As such, it can be concluded that 1,4-dioxane removal is still primarily occurring by ·OH oxidation and not by the other oxidizing/reducing species involved in e-beam treatment.

3.2. Bromate and other water quality data

Bromate samples were collected during some of the secondary effluent tests. Despite high bromide concentrations, 299 μg/L, and high ·OH radical exposures as shown above, no bromate formation was observed and all values were below detection. In the GAC effluent samples, downstream of ozonation, bromate removal of up to 82% was observed at a dose of 10.8 kGy with an initial concentration of 1.99 μg/L (Figure 4). Wang *et al.* (2016) showed that bromate removal was significantly lower with dissolved oxygen and nitrate present which is consistent with $e_{(aq)}^-$ scavenging by Equations (2) and (3) (Buxton 1988).

$$O_2 + e_{(aq)}^- \rightarrow O_2^- \qquad k = 1.1*10^9 M^{-1}s^{-1} \tag{2}$$

$$NO_3^- + e_{(aq)}^- \rightarrow NO_3^{2-} \quad k = 9.7*10^9 M^{-1}s^{-1} \tag{3}$$

Figure 5 | Effects of e-beam treatment on (a) dissolved oxygen, (b) nitrogen species, and (c) bulk organics in secondary effluent.

For one test, dissolved oxygen was measured in the sample holder before and after irradiation (Figure 5(a)). It is assumed that all tests were conducted with initial saturation concentrations of DO. Figure 5(b) shows the change in total and inorganic nitrogen species through e-beam irradiation. Nitrate concentrations increased at low doses and then decreased with increasing e-beam dose above 1.16 kGy. Doses much higher than those required for 1,4-dioxane were needed for significant nitrate reduction. Dissolved oxygen may have limited nitrate reduction as it would be the predominant $e^-_{(aq)}$ scavenger initially due to its higher initial concentration and slightly higher second order rate constant. Nitrite formation was observed at the lower doses but then also decreased at higher doses. Nitrate and nitrite were likely reduced to another inorganic nitrogen species which was not measured as the total nitrogen concentration did not decrease through irradiation. NO and N_2O formation seem unlikely as these two species are volatile and would likely be lost between sample transfer and storage as well as the air sparging step in the TOC/TN analysis. Ammonia concentrations increased to up to 0.75 mg/L-N, this is most likely release from dissolved organic nitrogen (rather than cell lysis as the samples were pre-filtered), which has been observed due to e^- transfer in ozonation (de Vera et al. 2017). A similar trend in nitrite and nitrate was observed in a second test shown in the SI at doses up to 2 kGy though ammonia formation was not as consistent and is worthy of further investigation.

Bulk organic matter was measured as dissolved organic carbon (DOC), chemical oxygen demand, and UV_{254} absorbance (Figure 5(c)). Overall, there was little change in the organics indicating that, similar to other advanced oxidation processes, complete mineralization of contaminants was not occurring. The slight increase in COD and then subsequent decrease could be due to reduction products being formed and then oxidized at higher doses. However, the slight increase in DOC at the lowest dose compared to the initial sample indicates that there may be some sample contamination, perhaps leaching of organics from the cork gasket used to seal the sample holders.

3.3. Applications and engineering significance

Electrical energy per order values (EEO) were calculated from the results above using Equation (4) (Londhe et al. 2021).

$$EEO_{e-beam} = \frac{Dose\ (kGy)}{3.6* - log_{10}\left(\frac{C}{Co}\right)} \tag{4}$$

Due to the non-linear relationship between log-removal and dose, EEOs were calculated using the results closest to 1-log removal in each water matrix. For secondary effluent, the average EEO value was reported from the different sample runs, excluding the samples with the background levels of 1,4-dioxane. The EEO values for 1,4-dioxane by electron beam were 0.37, 0.19, and 0.05 kWh/m³/order for secondary effluent, GAC effluent, and UPW, respectively. With a typical 70% transfer and generation efficiency applied the values were 0.53, 0.26, and 0.08 kWh/m³/order.

Ozonation has repeatedly been shown to be the most efficient AOP in wastewater effluent (Katsoyiannis et al. 2011; Lee et al. 2016; Sgroi et al. 2021). However, there is currently no available EEO data for 1,4-dioxane in secondary effluent. Lee

et al. (2016) calculated EEO values of 0.15 kWh/m^3/order for meprobamate and atrazine, which have similar second order rate constants with ozone and hydroxyl radicals to 1,4-dioxane. While this is significantly lower than the value here, it does not account for oxygen, or any chemicals required to suppress bromate formation, which was shown to be a significant challenge in this water (Pearce *et al.* 2022). UVAOP using hydrogen peroxide is the most common technique for 1,4-dioxane removal in drinking water treatment. An EEO of 0.13 kWh/m^3/order was calculated from a bench scale UV reactor treating 1,4-dioxane in deionized water with 10 mg/L H$_2$O$_2$, and 0.20 kWh/m^3/order on average for four groundwaters treated under the same conditions (Lee *et al.* 2020). Here e-beam treatment is comparable to UVAOP and again does not require any external reagents which may need to be quenched. Additionally, as discussed above, the non-uniform dose distribution of this experimental configuration may limit the efficiency of e-beam, especially when targeting removal of 90 percent or greater. The configuration of the sample holders used in this study did not allow for mixing of the samples which may have improved the dose distribution. Simply as an advanced oxidation process, e-beam treatment may be comparable in terms of operating costs to other conventionally used processes as discussed above. However, high capital cost of accelerators are an obstacle towards wider adoption of electron beam treatment (Miklos *et al.* 2018).

The clear benefit of e-beam treatment is the added reductive capacity when compared to traditional advanced oxidation processes. Certain compounds are more susceptible to degradation by reduction than oxidation. Acetone is primarily degraded by hydrated electrons (Yoon *et al.* 2009), whereas carbamazepine, bisphenol A (BPA), and atrazine are susceptible to both ·OH oxidation and e$^-_{(aq)}$/H· reduction (Basfar *et al.* 2009; Zheng *et al.* 2020; Trojanowicz *et al.* 2021). Certain halogenated contaminants and disinfection byproducts have been shown to be susceptible to radiolytic treatment and hydrated electron degradation such as trichloroethylene, TCE (Nickelsen *et al.* 2002), and carbon tetrachloride (Mak *et al.* 1997). The removal of PFAS by e-beam is the most promising. The higher doses required for PFAS degradation would guarantee that most other pollutants are well degraded as well. If e-beam treatment could be used for PFAS removal at doses <5 kGy then it may be a cost-effective alternative to the combination of ozonation and GAC in a wastewater treatment or water reuse context (Wang *et al.* 2016; Londhe *et al.* 2021).

While e-beam has been used in a large-scale, continuous flow application (Wang *et al.* 2022), there is limited water industry experience and few equipment vendors available for these uses. The development of high-power (several hundreds of kW), high-efficiency accelerators would allow the treatment of a higher water flow (tens of thousands of m^3/day) thereby reducing the impact of their capital cost to the overall treatment cost. R&D is ongoing at National Laboratories to develop such kind of accelerators (Ciovati *et al.* 2018; Dhuley *et al.* 2022). The results presented show that electron beam treatment is effective as an advanced oxidation process and for cost effective 1,4-dioxane removal, warranting further studies on the removal of other chemicals from water and wastewater, such as PFAS.

4. CONCLUSIONS

Electron beam treatment was evaluated for 1,4-dioxane removal in water and wastewater treatment applications. Greater than 90% removal was achievable at moderate doses and removal improved in the progressively cleaner water matrices. Using tert-butanol it was shown that 1,4-dioxane degradation primarily occurs through hydroxyl radical oxidation. Water radiolysis did not result in significant reductions in TOC or COD indicating that complete mineralization of contaminants was unlikely to have occurred. Bromate removal was observed in several samples and there was no bromate formation, despite the high bromide concentrations and ·OH exposures. The calculated EEO values for 1,4-dioxane removal in secondary wastewater effluent and GAC effluent were higher when compared to established treatment techniques in similar conditions though electron beam treatment would result in comparable operating costs in several cases. Some of this inefficiency may be due to experimental factors which warrants further evaluation at larger scales. Electron beam treatment can also be a promising method for treating PFAS compounds in wastewater and research on this topic has begun through the collaboration between HRSD and Jefferson Lab.

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FUNDING

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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