

# Perfluorooctanoic acid Degradation by UV/Chlorine

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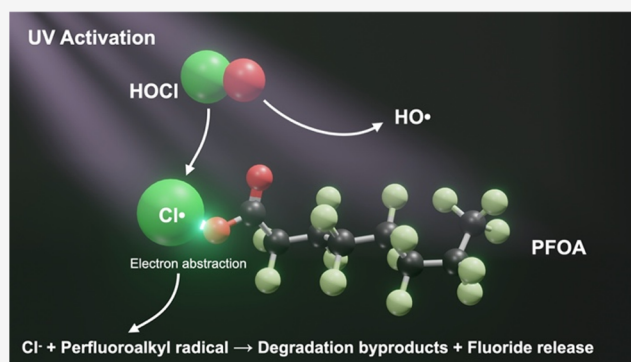
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Supporting Information

**ABSTRACT:** While per- and polyfluoroalkyl substances (PFAS) are recalcitrant to chemical reactions traditionally used in water treatment, we report the novel finding that combining ultraviolet (UV, 254 nm) light and chlorine can promote perfluorooctanoic acid (PFOA) degradation. About 12% removal of 100  $\mu\text{g/L}$  PFOA was observed after 30 min of irradiation ( $6.5 \times 10^{-6}$  Einstein  $\text{L}^{-1} \text{s}^{-1}$ ) in the presence of 1.4 mM (106 mg/L) NaOCl, compared to only 1% removal by UV photolysis and no removal by NaOCl alone. UV/chlorine with 0.02 mM NaOCl (1.5 mg/L, a more common dose for water treatment) removed 6  $\mu\text{g/L}$  PFOA within 30 min. To better detect defluorination, 50 mg/L PFOA was used, and UV/chlorine released significantly more fluoride (382  $\mu\text{g/L}$ ) than UV photolysis (0  $\mu\text{g/L}$ ) and dark controls (0  $\mu\text{g/L}$ ) over 30 min. By 60 min, this represents 32% of the maximum possible defluorination for the amount of PFOA removed by UV/chlorine versus 2% for UV photolysis. Radical scavenger tests indicated that  $\text{Cl}^\bullet$  and  $\text{Cl}_2^{\bullet-}$  play a crucial role in PFOA degradation, which we postulate is initiated by electron abstraction leading to a decarboxylation–hydroxylation–elimination–hydrolysis pathway. Whereas reaction rates were relatively slow for practical application in water treatment plants, these results underscore overlooked reactions with common water treatment constituents that may influence the fate of PFAS.

**KEYWORDS:** PFAS, PFOA, photolysis, water treatment, degradation, UV/chlorine



## INTRODUCTION

Per- and polyfluoroalkyl substances (PFAS) are a class of over 4700 compounds<sup>1</sup> widely used in industry that have been linked to adverse health outcomes after prolonged exposure and bioaccumulation,<sup>2,3</sup> including immunotoxic effects and increased cancer risk.<sup>4,5</sup> The widespread use of PFAS has led to dissemination in water systems around the world,<sup>6</sup> which represents a costly problem for water service providers.<sup>7–9</sup> Traditional water treatment approaches such as granular activated carbon (GAC) filtration used by most utilities only remove PFAS without destroying it, and regeneration or replacement of GAC beds is often required at least once a year.<sup>10,11</sup> Incineration, the most widely used PFAS destruction technique, is expensive because of the high temperatures (>1000 °C) needed to degrade many PFAS and is not feasible for water treatment systems.<sup>12,13</sup> New technologies are required to aid water and wastewater treatment facilities in degrading PFAS.

Various PFAS treatment methods have been considered over the past few years, often focusing on perfluorooctanoic acid (PFOA), one of the most abundant and widely studied PFAS. Many approaches to degrade hazardous contaminants rely on advanced oxidation processes (AOPs),<sup>14</sup> such as photocatalysis,<sup>15,16</sup> sonolysis,<sup>17,18</sup> Fenton-based processes,<sup>19,20</sup> as well as advanced reduction processes.<sup>21,22</sup> Ultraviolet (UV) light is often used for AOPs, so UV-activated treatments could

be integrated into water or wastewater treatment facilities that already have UV disinfection in place. One of the simplest AOPs is UV/H<sub>2</sub>O<sub>2</sub>, which generates hydroxyl radicals (HO•) in solution to degrade many organic pollutants.<sup>23–25</sup> However, HO• is ineffective at initiating PFOA degradation.<sup>26</sup>

Several PFAS treatment methods that utilize other radicals have been proposed to degrade PFOA, and usually involve attacking weaker parts of the molecule, such as the  $\alpha$ -carbon by the terminal headgroup.<sup>27</sup> Sulfate radicals degrade PFOA at low pH values,<sup>28</sup> both through heat,<sup>29</sup> and UV activation.<sup>30</sup> UV-activated carbonate radical anions were also reported to slowly degrade PFOA,<sup>31</sup> while superoxide radicals were ineffective.<sup>32</sup> However, these UV-activated technologies have not been implemented at a wide scale in water treatment facilities because of the requirement of relatively long contact times,<sup>33</sup> and because of scavenging of reactive species by suspended solids, bicarbonate, and chloride ions found in natural waters.<sup>34,35</sup>

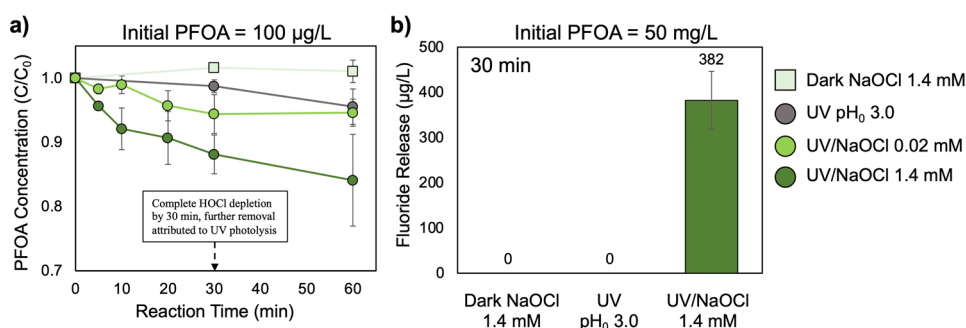
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**Figure 1.** UV/chlorine degrades and defluorinates PFOA to a greater extent than UV/photolysis over a 60 min reaction. These data show (a) PFOA removal, initial PFOA 100 µg/L, with two doses of NaOCl (1.4 mM (106 mg/L) or 0.02 mM (1.5 mg/L)), and UV photolysis control; (b) fluoride release at 30 min from reactions with 50 mg/L initial PFOA, NaOCl dose 1.4 mM (106 mg/L), and controls (LOD 10 µg/L for F<sup>-</sup>). All pH values were adjusted to pH 7 at the start of the reaction with NaOH or HClO<sub>4</sub>. Final pH was 3.0 (UV/NaOCl 1.4 mM) and 4.9 (UV/NaOCl 0.02 mM). Irradiation intensity =  $6.5 \times 10^{-6}$  Einstein L<sup>-1</sup> s<sup>-1</sup> (6.4 mW/cm<sup>2</sup>). Error bars represent  $\pm$  one standard deviation from the mean of triplicate reactors.

UV/chlorine treatment has received minimal attention for PFAS degradation,<sup>36,37</sup> because separately, these components are not effective at degrading PFAS.<sup>38–41</sup> While most PFAS (including PFOA) do not react with free chlorine, UV photolysis requires hours-long contact times for PFAS degradation compared to seconds for disinfection. However, notwithstanding concern about chlorinated organic byproducts generation,<sup>42–44</sup> combined UV/chlorine could offer a simple intervention using commonly available technology at a relatively low cost. UV/chlorine has been tested at the pilot-scale<sup>45,46</sup> to remove various contaminants such as atrazine,<sup>47</sup> 1,4-dioxane,<sup>45</sup> pharmaceuticals and personal care products (PPCPs),<sup>48</sup> and trichloroethylene<sup>49</sup> (which often co-occurs with PFAS in groundwater<sup>50</sup>), yet this technology has not been thoroughly investigated for PFAS degradation. The increasing use of this combined technology warrants further research on its merits and limitations in degrading PFAS, which is the purpose of this paper. We also discern the reactive species responsible for PFOA degradation and address how reaction conditions affect PFOA degradation and defluorination.

## MATERIALS AND METHODS

Chemicals, reagents, and sources are detailed in Text S1.

**UV/Chlorine Experiments with PFOA.** Sodium hypochlorite pentahydrate powder was added to deionized water (DI) to create a stock solution and stored in the dark at 4 °C. PFOA degradation experiments were conducted in a 20 mL solution in a 100 mL quartz flask containing DI, PFOA, and a Teflon-coated stir bar. Control tests demonstrated no fluoride release from stir bars (Table S3). PFOA initial concentrations were 100 µg/L (0.24 µM), on the high side of the range reported for natural waters (usually ng/L)<sup>41,51</sup> but not unprecedented for contaminated surface and groundwater sites.<sup>52,53</sup> Some tests were also conducted with 50 mg/L PFOA (120 µM), similar to previous research,<sup>54</sup> to facilitate analysis of released fluoride. NaOCl initial concentrations were 0.02 mM (1.5 mg/L) or 1.4 mM (106 mg/L). The latter value is very high for municipal water treatment but within the range used by other mechanistic studies.<sup>55–57</sup>

The solution pH was measured using a pH probe (Thermo Scientific Orion Versa Star Pro Benchtop pH Meter) and adjusted to pH 7 (unless otherwise stated) using HClO<sub>4</sub> or NaOH. For radical quencher experiments, 10 mM NaHCO<sub>3</sub> or 100 mM *tert*-butyl alcohol was also added to the flask, which was similarly adjusted to pH 7 (unless otherwise stated). A

rubber stopper sealed the reaction vessels. The flasks were covered with aluminum foil for dark reactions or before being placed into the UV reactors, which were previously described.<sup>58</sup> Briefly, the flask was placed on a stir plate inside a reactor where six 4W UVC (254 nm) bulbs (Ushio G4T5) irradiated the flask from two sides. The reactor was turned on for at least 30 min before each reaction to allow the UV flux to stabilize. Chemical actinometry was conducted using a standard potassium ferrioxalate method (Text S2)<sup>59</sup> to determine the photon flux, yielding  $6.5 \times 10^{-6}$  Einstein L<sup>-1</sup> s<sup>-1</sup> (6.4 mW/cm<sup>2</sup>).

Aliquots taken at each time point were not quenched with sodium sulfite (used to quench remaining HOCl/ClO<sup>-</sup>) unless explicitly stated, given that no PFOA degradation was observed with NaOCl in the dark, and sodium sulfite interfered with fluoride measurements.

**UV/H<sub>2</sub>O<sub>2</sub> Experiments.** The 30% H<sub>2</sub>O<sub>2</sub> was diluted and added to DI water with 100 µg/L PFOA, and the pH was adjusted to 7 or 3 with HClO<sub>4</sub> or NaOH. Reactions were run in a sealed quartz flask, as described.

**Radical Probe Experiments.** Radical production in the UV/NaOCl system was determined using nitrobenzene (HO•) and benzoic acid (HO•, Cl•) under normal reaction conditions, and aliquots were quenched with sodium sulfite (Text S3).

**Analytical Methods.** Analytical methods are described in the Supporting Information for PFOA (Text S4, S5), nitrobenzene and benzoic acid (Text S4), F<sup>-</sup> and Cl<sup>-</sup> (Text S6), and NaOCl titration (Text S7).

All experiments were conducted in triplicate. Student's *t* test (two-tailed, unpaired, assuming equal variance) was used to determine if results were significant at the 95% confidence level ( $p \leq 0.05$ ).

## RESULTS AND DISCUSSION

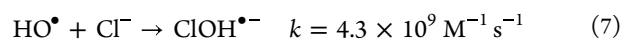
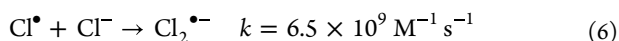
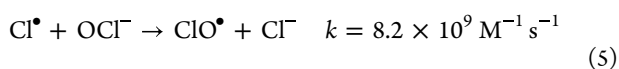
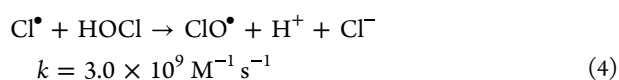
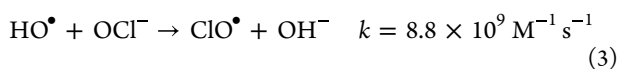
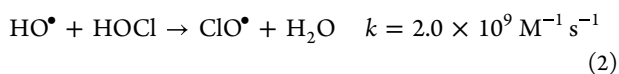
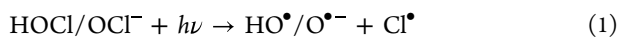
**UV/chlorine degrades PFOA to a significantly greater extent than UV photolysis.** Figure 1 demonstrates significant PFOA degradation and fluoride release over 1 h by UV/chlorine treatment, but not by NaOCl in the dark. Some degradation was also observed by UV photolysis (254 nm radiation at  $6.5 \times 10^{-6}$  Einstein L<sup>-1</sup> s<sup>-1</sup> [6.4 mW/cm<sup>2</sup>]), though to a much lower extent. These experiments were conducted in unbuffered solutions because running these reactions in 10 mM phosphate buffer at pH 7 (or pH 3 or 5) resulted in no degradation or fluoride release (Figure S1).

Previous studies reported a small pH effect on UV photolysis of PFOA, slightly favoring acidic pH for PFOA removal.<sup>60</sup> The pH values for the UV photolysis controls were chosen based on the initial (pH 7) and final (pH 3) values of the UV/chlorine system.

While UV/chlorine systems produce both hydroxyl radicals and reactive chlorine species, prior studies have shown that HO• cannot initiate PFOA degradation.<sup>26,61</sup> Here, we report the unprecedented finding that, at a representative 100 µg/L initial PFOA concentration ( $C_0$ ), UV/chlorine yielded a significantly lower residual PFOA concentration after 30 min ( $C_{30}$ ) than controls (Figure 1a). This indicates that reactive chlorine species can degrade PFOA. At an initial dose of 1.4 mM [106 mg/L] chlorine, the residual  $C_{30}/C_0$  value was  $0.88 \pm 0.03$ , compared to  $0.99 \pm 0.01$  for UV photolysis ( $p < 0.02$ ). Interestingly, with a lower chlorine dosage (0.02 mM [1.5 mg/L]), representing a common chlorine dosage applied for disinfection in water treatment facilities, UV/chlorine also degraded about 6% of the added PFOA in the first 30 min ( $p < 0.04$ ). This demonstrates that chlorine concentrations used in water treatment could remove some PFOA under UV irradiation. Additionally, NaOCl solution (1.4 mM [106 mg/L]) at pH 7 without UV irradiation (dark control) did not degrade PFOA.

Figure S2 shows that, at an uncommonly high PFOA concentration (50 mg/L, utilized to more easily detect fluoride release), UV photolysis and UV/NaOCl removed similar levels of PFOA over 1 h, with no detectable PFOA degradation in the first 30 min. However, the released fluoride concentrations were significantly higher for the UV/chlorine treatment ( $382 \pm 64$  µg/L) than for UV photolysis at pH<sub>0</sub> 3.0 ( $0.0 \pm 0.0$  µg/L) and the dark NaOCl control ( $0.0 \pm 0.0$  µg/L) at 30 min (Figure 1b). This demonstrates a greater potential of UV/chlorine to defluorinate PFOA than UV photolysis.

**Radical Scavenging Experiments Suggest the Key Role of Cl• in PFOA Degradation.** The radicals produced by the UV/chlorine process were explored to understand how UV/chlorine degrades and defluorinates PFOA. UV/chlorine creates many radicals, principally a hydroxyl radical and a chlorine radical, which can undergo secondary reactions to produce reactive chlorine species (RCS). While hundreds of RCS reactions have been explored,<sup>48,62</sup> the major initial radicals include HO•, Cl•, Cl<sub>2</sub>•<sup>-</sup>, ClO•, and ClOH•<sup>-</sup>, which can be produced from chlorine photolysis at wavelengths below 400 nm.<sup>63,64</sup> Radical formation and corresponding rate constants are shown in eqs 1–7.<sup>42,65</sup> Ozone formation was not considered as it is negligible at 254 nm.<sup>36,44,66</sup>



The relative abundance of these radicals depends on reaction conditions, especially the pH. At neutral pH, HO• and Cl<sub>2</sub>•<sup>-</sup> predominate, but as the pH decreases, both Cl• and Cl<sub>2</sub>•<sup>-</sup> concentrations increase, and HO• decreases in relative abundance.<sup>42</sup> Additionally, the quantum yield of HOCl photolysis increases with decreasing pH, further enhancing radical formation.<sup>42</sup> Given that this system was unbuffered, the solution pH decreased as the reaction progressed, reaching pH 3.2 by 5 min (Table S4). Thus, most of the degradation occurred under acidic conditions. This led to increased chlorine radical formation, based on the law of mass action and the equilibrium equations of the different RCS reactions, as previously described.<sup>42,65,67,68</sup> Radical probe studies yielded a steady state concentration of [HO•]<sub>ss</sub> =  $1.4 \times 10^{-12}$  M and [Cl•]<sub>ss</sub> =  $5.8 \times 10^{-13}$  M (Text S3, Figure S3), within the range of commonly calculated values.<sup>36</sup> The role of these radicals was explored to further understand the PFOA degradation mechanism.

Radical scavenger tests were conducted to determine the role of different radicals (Figures S4 and S5). Bicarbonate is more selective for some RCS (Cl• =  $2.2 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$ , Cl<sub>2</sub>•<sup>-</sup> =  $8.0 \times 10^7 \text{ M}^{-1} \text{ s}^{-1}$ ) than others (ClO• =  $600 \text{ M}^{-1} \text{ s}^{-1}$ ),<sup>69,70</sup> and less selective for HO• ( $8.6 \times 10^6 \text{ M}^{-1} \text{ s}^{-1}$ ).<sup>71</sup> *tert*-Butyl alcohol (TBA) is often used as a hydroxyl radical scavenger ( $6.0 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$ ),<sup>71</sup> however, it can also scavenge prominent RCS at similarly high rates, with Cl• ( $3.0 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$ ) and ClO• ( $1.3 \times 10^7 \text{ M}^{-1} \text{ s}^{-1}$ ), though much less for Cl<sub>2</sub>•<sup>-</sup> ( $700 \text{ M}^{-1} \text{ s}^{-1}$ ).<sup>69,70</sup> NaHCO<sub>3</sub> addition (10 mM) halted PFOA removal and yielded no detectable defluorination (Figure S4). This is likely because of both scavenging and the higher pH, which did not decrease below 7 because of bicarbonate's buffering effect, though experiments with initial pH 5 or pH 3 also showed no PFOA removal or detected fluoride release (Figure S4). This suggests that ClO•, with its lower oxidation potential (Table S5), did not play a significant role. Additionally, bicarbonate ions can react with RCS to produce carbonate radical anions, CO<sub>3</sub>•<sup>-</sup>, which may degrade PFOA.<sup>31</sup> However, no defluorination was observed in this system, so this should not have caused interference.

TBA (100 mM) significantly decreased fluoride release (0 µg/L) at 30 min compared to UV/NaOCl without scavengers ( $382 \pm 64$  µg/L) (Figure S6). For tests with 100 µg/L PFOA, its removal was significantly impaired ( $p < 0.02$ ) but was still appreciable in the first 30 min (Figure S6). TBA scavenges HO• and Cl• but not Cl<sub>2</sub>•<sup>-</sup>.<sup>69,70</sup> The small but statistically significant PFOA removal ( $p < 0.03$ ) in the TBA-amended system suggests that Cl<sub>2</sub>•<sup>-</sup> could initiate PFOA degradation; however, the rate was slow, likely because of TBA's scavenging of Cl•, which is a precursor for Cl<sub>2</sub>•<sup>-</sup> formation (eq 6). Additionally, PFOA removal and defluorination were completely quenched during tests with 10 mM NaHCO<sub>3</sub> (which quickly scavenges both Cl• and Cl<sub>2</sub>•<sup>-</sup>), providing further evidence for the role of these RCS. Cl<sub>2</sub>•<sup>-</sup> formation is enhanced by the presence of Cl<sup>-</sup>, based on the law of mass action (eq 6). Figure S6 shows an insignificant difference in PFOA removal with 10 mM NaCl added ( $p > 0.1$ ). However, background chloride (~3 mM) present from reaction precursors (Figure S7) apparently promoted Cl<sub>2</sub>•<sup>-</sup> formation such that the addition of more salt did not accelerate PFOA removal. Overall, scavenger studies indicate a primary role of

$\text{Cl}^\bullet$ , with a potential secondary role of  $\text{Cl}_2^{\bullet-}$  for PFOA removal.

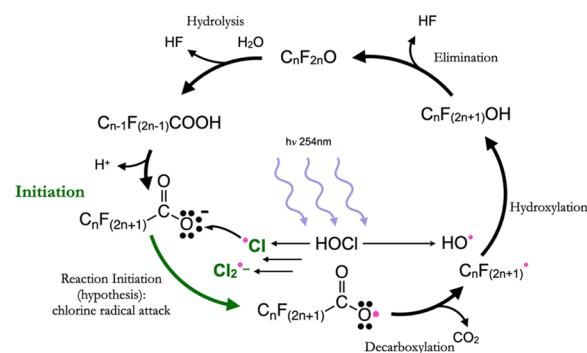
UV/ $\text{H}_2\text{O}_2$  experiments were conducted to confirm that hydroxyl radicals do not initiate PFOA degradation, as previously shown.<sup>26</sup> Using 100  $\mu\text{g}/\text{L}$  PFOA and either 1.4 mM (48 mg/L) or 148 mM (5 g/L)  $\text{H}_2\text{O}_2$ , no significant PFOA degradation was observed compared to UV photolysis (Figure S8). This corroborates the scavenger test results, suggesting that  $\text{Cl}^\bullet$  and  $\text{Cl}_2^{\bullet-}$  (with relatively high reduction potentials) play a key role in initiating PFOA degradation.

Free chlorine was photolyzed and consumed within 30 min (Figure S9), which is reflected by the faster initial PFOA degradation rate (Figure 1a). RCS should no longer be generated once free chlorine is expended (eq 1). Because  $\text{HO}^\bullet$  cannot initiate PFOA degradation<sup>26,61</sup> (Figure S8), subsequent PFOA removal was likely because of UV photolysis, which is much slower. This demonstrates the critical role of RCS in PFOA degradation.

**PFOA Degradation by UV/Chlorine likely Follows DHEH Mechanism.** One commonly discussed degradation mechanism for PFOA degradation by AOPs is the decarboxylation–hydroxylation–elimination–hydrolysis (DHEH) pathway.<sup>27,61,72,73</sup> The initial oxidation step requires the abstraction of an electron from the terminal carboxyl group, which is usually in the deprotonated form because of PFOA's low  $\text{p}K_a < 1.3$ .<sup>74–77</sup> Chlorine radicals are powerful oxidants and are known to react with carboxylic acids more selectively than hydroxyl radicals.<sup>36</sup> Thus, we postulate that chlorine radicals could remove an electron from  $\text{C}_7\text{F}_{15}\text{COO}^-$ , forming  $\text{C}_7\text{F}_{15}\text{COO}^\bullet$  and initiating the DHEH mechanism. The standard reduction potentials of  $\text{Cl}^\bullet$  and  $\text{Cl}_2^{\bullet-}$  are +2.432 and 2.126 V, respectively (Table S4).<sup>78</sup> This is a UV-activated radical mechanism in a homogeneous system; a similar reaction mechanism was shown for UV-activated  $\text{CO}_3^{\bullet-}$  (one-electron reduction potential of 1.78 V at pH 7), which initiated PFOA degradation by abstracting an electron from the perfluorinated carboxyl anion.<sup>31</sup>  $\text{CO}_3^{\bullet-}$  is a strong oxidant; however, it has a lower reduction potential than  $\text{Cl}^\bullet$  and  $\text{Cl}_2^{\bullet-}$ , suggesting that these RCS are viable reactants for PFOA degradation.

Figure 2 proposes a UV/chlorine assisted DHEH mechanism. Attack by  $\text{Cl}^\bullet/\text{Cl}_2^{\bullet-}$  would lead to decarboxylation because of the instability of  $-\text{COO}^\bullet$ , forming a perfluoroalkyl radical (Figure 2). Hydroxyl radicals could then react with this perfluoroalkyl radical to create a perfluoroalcohol, which is unstable in solution and would undergo hydrogen fluoride elimination and hydrolysis.<sup>61,73</sup> This would regenerate a carboxylic acid with one fewer carbon, and the reaction would begin again. TBA inhibited defluorination but not PFOA removal, suggesting that  $\text{Cl}_2^{\bullet-}$  can initiate PFOA degradation, but  $\text{HO}^\bullet$  scavenging by TBA apparently precludes the DHEH cycle from efficiently proceeding to the fluorine elimination step.

To rule out the generation of chlorine-substituted PFOA byproducts, high-performance liquid chromatography tandem mass spectrometry analysis was conducted in MS Scan mode (Text S5). None were found (limit of detection (LOD) of 1  $\mu\text{g}/\text{L}$ ) within the search parameters. While this does not rule out the possibility of chlorinated PFAS byproducts or a substitution mechanism, the lack of evidence of these products and the demonstration of perfluorocarboxylic acid degradation byproducts, primarily PFHpA and PFHxA (Figure S10),



**Figure 2.** Schematic of DHEH pathway for PFOA degradation using chlorine radicals and dichlorine radicals as the activating species to initiate degradation in the UV/chlorine system.  $\text{Cl}^\bullet$  and  $\text{Cl}_2^{\bullet-}$  are the primary species that are predicted to initiate PFOA degradation, while  $\text{HO}^\bullet$  is utilized at a later step for perfluoroalcohol formation, which is unstable in solution and degrades, releasing fluoride. The compound is reoxidized, and the cycle starts again with one fewer carbon and two fewer fluorine atoms in the chain.

indicates a sequential decarboxylation mechanism consistent with the DHEH pathway.

**Implications.** UV/chlorine treatment resulted in unprecedented defluorination and PFOA removal compared to UV treatment and chlorination controls. However, rates were relatively slow in the context of typical retention times in UV irradiation and chlorination processes used in water or wastewater treatment plants. In fact, the high chlorine concentrations, long irradiation times, and acidic conditions used here are atypical of normal treatment conditions. Nevertheless, given the widespread availability of UV and chlorination disinfection infrastructure and the increase in research and pilot studies considering UV/chlorine treatment,<sup>45,46</sup> these results provide timely mechanistic insight into overlooked reactions that may affect the fate of PFAS in drinking water or wastewater treatment systems. Whether these findings are also relevant to remediation of groundwater or other water sources impacted by ng/L PFAS levels would require further studies and techno-economic analysis.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.estlett.2c00452>.

Additional methods, materials, and experimental details referenced in the text (PDF)

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## Notes

The authors declare no competing financial interest.

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