EXECUTIVE SUMMARY

Ex Situ Remediation of Investigation-Derived Wastes containing PFAS by Electron Beam Technology

SERDP Project ER18-1620

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ACRONYMS AND ABBREVIATIONS

AFB	Airforce Base
eBeam	Electron beam
kGy	kilogray
kW	kilowatt
MeV	million electron volts
IDW	Investigation-derived wastes
NASJRB/WG	Naval Air Station Joint Reserve Base Willow Grove
PFAS	per and polyfluoroalkyl substances
PFCA	perfluorinated carboxylic acids
PFOS	perfluorooctane sulfonate
PFOA	perfluorooctanoic acid
PFSA	Perfluorosulfonic acids

ACKNOWLEDGEMENT

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1.0 INTRODUCTION

Electron beam (eBeam) technology utilizes electron accelerators to generate extremely large numbers of highly energetic electrons from electricity. Compact, high-energy (10 million electron volts, MeV) and high power (~ 500 kilowatts, kW) accelerators capable of treating upwards of 1 million gallons per day of groundwater are commercially available today. These accelerators can produce extremely large concentrations of highly energetic electrons (> 10^{15} electrons/cm^2/sec) when they interact with water produce extremely large amounts of highly reactive free radicals ($H^{\bullet}$, e^-, and $HO^{\bullet}$), hydrogen peroxide (H_2O_2), hydrogen atoms (H_2), and hydrated protons (H_3O^+). Electron beam as a remediation technology is paradigm shifting because it simultaneously employs both reduction and oxidation processes. At very high doses, temperature is also involved in these complex reactions. These powerful oxidation-reduction reactions occur almost instantaneously and are therefore best characterized as an *Advanced Oxidation-Reduction Process.* Since this ionizing technology relies on commercial electricity, this is a "switch- on/switch-off" technology without the need for radioactive materials. The proposed research was directly aligned with the scope of SERDP's SEED project which is to explore innovative approaches that entail high technical and scientific risk.

2.0 OBJECTIVES

The specific objectives of the SERDP Exploratory Development (SEED) project were,

1. To quantify the effectiveness of eBeam technology at breaking down perfluorooctane sulfonate (PFOS) in soil and water under controlled experimental conditions, and
2. To characterize the effectiveness of eBeam at destructive treatment for a mixture of per and polyfluoroalkyl substances (PFAS) present in actual Investigation-derived wastes (IDW) samples collected from multiple sites.

3.0 TECHNICAL APPROACH

The project was divided into specific experimental tasks. In Task 1, the experiments focused on exposing PFOS and PFAS spiked sand and aqueous samples at varying eBeam doses to identify the optimal dose range for treatment. Task 2 focused on demonstrating the efficacy of eBeam technology for the breakdown of PFOS and perfluorooctanoic acid (PFOA) and other PFAS in actual IDW samples obtained from Pennsylvania (Willow Grove NASJRB/WG) and Michigan (Wurtsmith Air Force Base [AFB]). Task 3 focused on analyzing the economic feasibility of treatment by identifying the capital and operating costs for treating PFAS-contaminated IDW soils.

In **Task 1**, the project team evaluated the effectiveness of varying eBeam low doses (10 kGy – 500 kGy) as well as experimental conditions such as alkalinity, nitrogen sparging, and nitrate amendments on its ability to degrade PFOS and PFOA. PFAS-free laboratory sand (10 g) and distilled water were spiked with PFOS (10µg/L and 20 mg/kg sand) and PFOA (5 µg/L and 10 mg/kg sand). These batch-scale studies were performed in PFAS-free 60 mL square-sided HDPE bottles using the 10 MeV S-band 15 kW eBeam linear accelerator at the National Center for Electron Beam Research at Texas A&M University. Industry-standard, internationally traceable alanine dosimeters were used to measure the absorbed eBeam doses.

The samples were exposed to 50 kGy and 500 kGy eBeam doses. The 500 kGy dose was delivered incrementally. The control (untreated) and the eBeam-exposed treated samples were analyzed for PFOA and PFOS using a commercial analytical laboratory (SGS-AXYS) as well as an in-house university laboratory.

In **Task 2**, the focus was on delivering eBeam doses to actual PFAS-contaminated IDW groundwater and soil samples. Compared to delivering 50 kGy to experimental samples in HDPE bottles, the delivery of higher doses required the fabrication of custom-designed experimental vessels (Fig. ES-1). The experimental vessel consisted of a sealed aluminum pressure vessel that was connected to a condensate collection vessel. The samples were placed in a "boat" within the experimental sealed pressure vessel using stainless steel foil. Condensate collection vessels and thermocouples were employed. The dose rate of the linac was used to calibrate the delivered doses. In these experiments, we also evaluated the value of amendments such as $NaNO_3$, $NaHCO_3$, pH adjustments (to alkaline conditions), and moisture content on PFOS degradation. The IDW samples were obtained from Pennsylvania (Willow Grove NASJRB/WG) and Michigan (Wurtsmith AFB). Portions of these samples were sent to the commercial laboratory for analysis per the EPA Method 537.

We focused the studies on the Willow Grove (PA) and Wurtsmith (MI) IDW samples since they had high concentrations of PFOS in the groundwater and soil respectively (Table ES-1). The samples were exposed to high doses (500 kGy, 1000 kGy, and 2000 kGy). To confirm the reproducibility of the high dose eBeam results of the IDW soil samples, the project team repeated these studies using three replicate sample "boats" within the treatment chamber. The Michigan IDW soil samples were dried to 10% soil moisture content and 30g -50 g were employed in these confirmation studies.

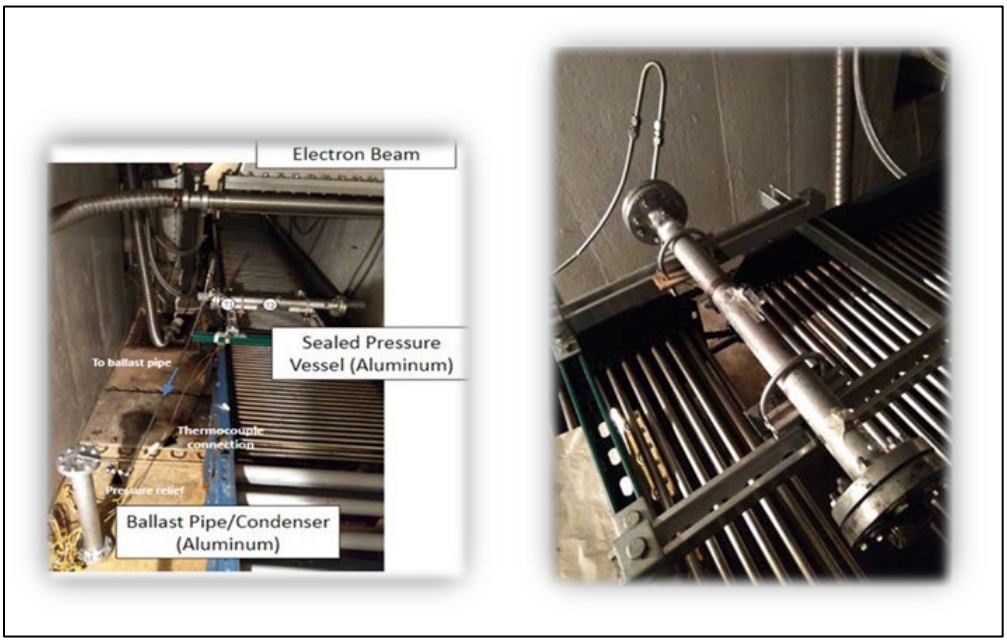


Figure ES-1. Photos Showing the Experimental Vessels Used for High Dose eBeam Treatments

Table ES-1. PFAS Concentration in Investigation – Derived Waste Groundwater and Soil Samples from Pennsylvania and Michigan

	Field IDW Sample Michigan Soil	Field IDW PA Soil	Field IDW Michigan Water	Field IDW PA Water
	NG/G	NG/G	NG/L	NG/L
PFBA	6.91	<0.269	23.1	<328
PFPeA	33.9	0.155	88.4	402
PFHxA	11.1	0.15	53.4	1250
PFHpA	13.1	<0.067	19.2	218
PFOA	**17.2**	**0.098**	**41.6**	**870**
PFNA	2.98	<0.067	3.97	<80
PFDA	0.964	<0.067	<3	<80
PFUnA	<0.25	<0.067	<3	<80
PFBS	1.16	<0.062	<3.1	505
PFPeS	0.717	0.069	<3	664
PFHxS	29.2	0.833	84.7	4240
PFHpS	3.94	<0.067	8.18	760
PFOS	**1010**	**8.3**	**1370**	**42600**
PFNS	1.66	<0.067	<3	87.3
PFDS	<0.25	<0.067	<3.1	<80
6:2 FTS	13.1	1.12	138	1080
8:2 FTS	30.5	<0.262	67.9	<320
PFOSA	124	<0.067	70.3	166
% Moisture	84.4	6.94		

Task 3 consisted of an economic analysis for use of the eBeam technology to remediate PFAS-contaminated soil samples at 1500 kGy. Capital and operating costs of a fixed eBeam technology platform were included in this analysis.

4.0 RESULTS AND DISCUSSION

Figures ES-2A and ES-2B show that at 50 kGy, both PFOS and PFOA are degraded in the spiked distilled water samples. At 50 kGy, as much as 87% of PFOA was degraded. At 50 kGy, only 16% of PFOS was degraded in the aqueous samples. The PFOA and PFOS reductions were, however, statistically significant ($p<0.05$) when comparing the control and eBeam treated samples. Figures ES-3A and ES-3B show the results when PFOA and PFOS spiked sand samples are exposed to 50 kGy. At this dose, as much as 86% of PFOA was degraded while only 27.5% of PFOS was degraded in the sand samples. These reductions were statistically significant ($p<0.05$) when comparing the control and eBeam-treated sand samples. Figure ES-4 shows that as the eBeam dose is increased from 50 kGy to 250 kGy, there was not a statistically significant ($p> 0.05$) increase in PFOS breakdown. However, when the dose increased from 250 kGy to 500 kGy, there was a statistically significant difference ($p<0.05$) in the reduction of PFOS in the PFOS-spiked sand samples. At 500 kGy, PFOS concentration in the spiked sand samples was reduced by 41.4%.

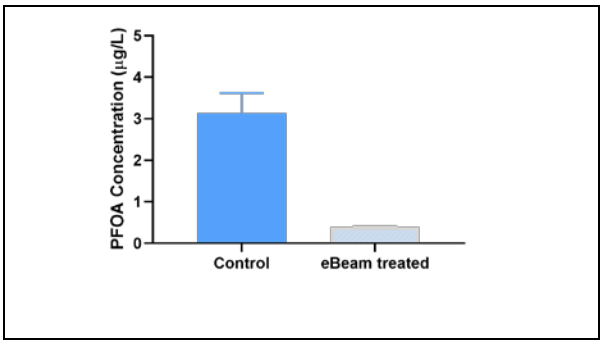


Figure ES-2A. eBeam-mediated (50 kGy) Degradation of PFOA in Laboratory Spiked Water Samples

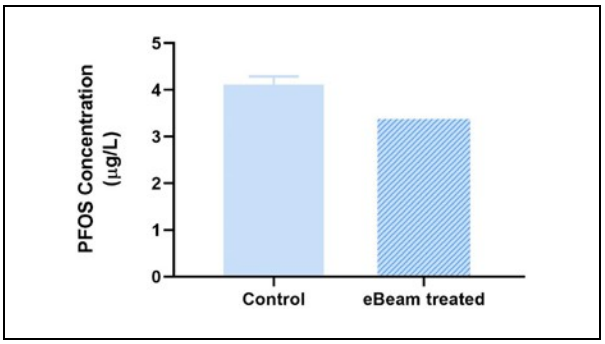


Figure ES-2B. eBeam-mediated (50 kGy) Degradation of PFOS in Laboratory Spiked Water Samples

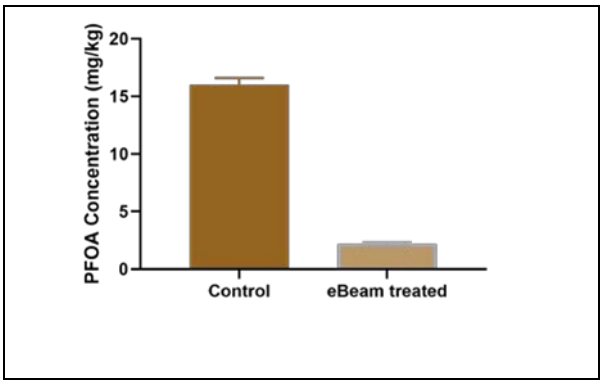


Figure ES-3A. eBeam-mediated degradation of PFOA in laboratory spiked sand samples

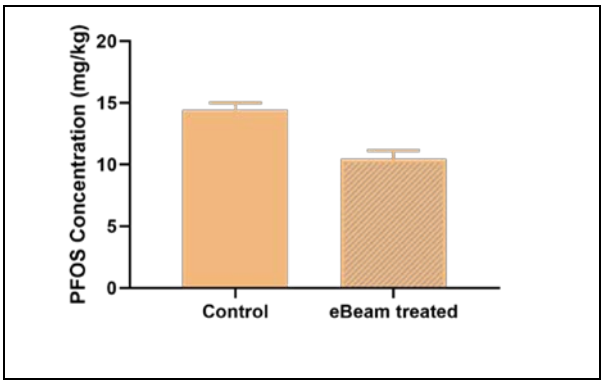


Figure ES-3B. eBeam-mediated degradation of PFOS in laboratory spiked sand samples

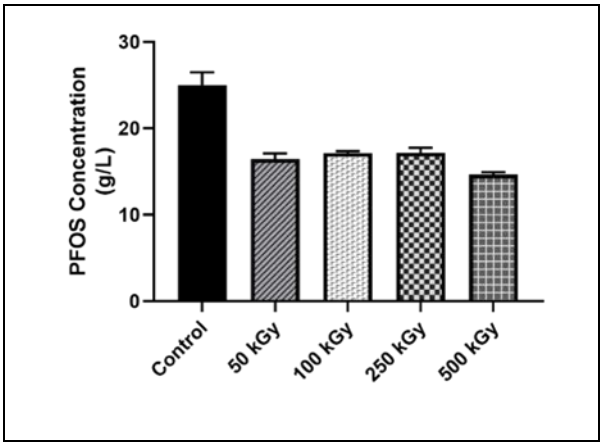


Figure ES-4. Effect of Increasing eBeam Doses on PFOS Degradation in Laboratory-spiked Water Samples

The results from higher eBeam doses of the laboratory–spiked water and sand samples are shown in Figure ES-5 and Figure ES-6. At 500 kGy, there was a 48.6% reduction in PFOS concentration (in the absence of amendments such as pH adjustment to 13, $NaNO_3$ and $NaHCO_3$) as compared to 22% reduction in the presence of amendments. At 2000 kGy, however, there was 88.8% PFOS reduction (in the presence of amendments) as compared to 96.6% reduction in the absence of amendments (Fig ES-5). The reduction in the sand samples were even larger. At 500 kGy, there was a 98.8% reduction in PFOS concentration in the absence of amendments as compared to greater than 99.99% reduction at 2000 kGy (Fig. ES-6). These results suggested that eBeam-mediated breakdown of PFOA and PFOS was significantly better in sand samples as compared to aqueous samples.

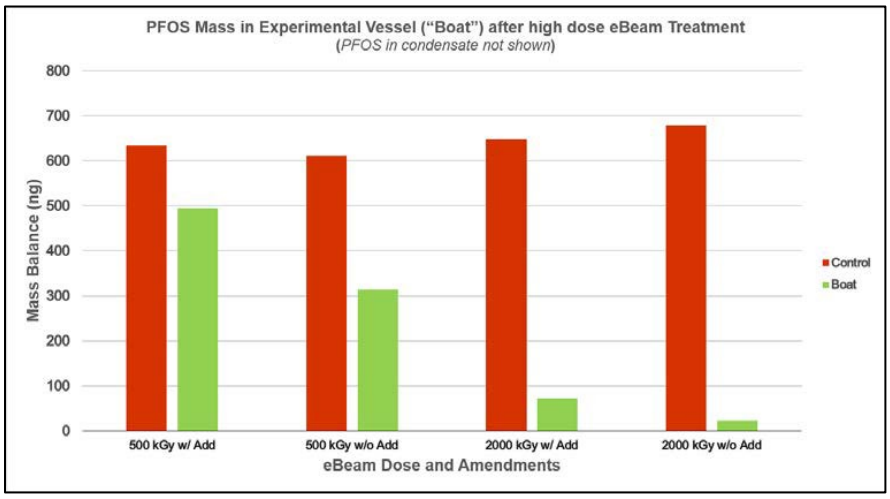


Figure ES-5. PFOS Degradation in Laboratory-spiked Water Samples as a Function of Increasing eBeam Doses.

(Amendments refer to pH, NaNO3 and NAHCO3 adjustments)

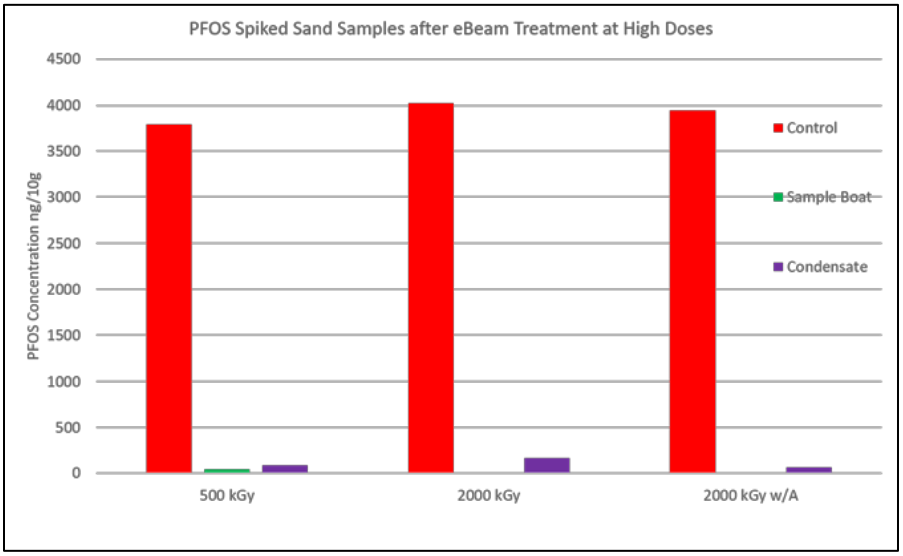


Figure ES-6. PFOS Degradation in Laboratory-spiked Sand Samples as a Function of Increasing eBeam Doses.

(Amendments refer to pH, NaNO3 and NAHCO3 adjustments)

The results from the exposure of IDW groundwater from Pennsylvania (Willow Grove NASJRB/WG) to high eBeam doses are shown in Table ES-2. The untreated and eBeam irradiated Willow Grove groundwater samples were analyzed by the commercial laboratory using the post analysis TOP assay. The results indicate that eBeam technology at high doses can be effective at degrading PFOS. At 2000 kGy, an 87.9% reduction of PFOS concentration was observed. The PFOA concentration increased during the eBeam exposure. This suggests that PFOA is accumulating during the breakdown of PFOS. The accumulation of PFOA as a breakdown product of PFOS has been recently reported in the literature.

Table ES-2. eBeam-mediated reakdown of PFOA and PFOS in Willow Grove (PA) IDW Groundwater Samples.

The % removal on a mass basis is shown.

	Treatment	Post-TOP PFOA in boat (ng)	Post-TOP PFOS in boat (ng)	% Removal of Post-TOP PFOA	% Removal of Post-TOP PFOS
PA-IDW-GW	**0 kGy**	112	3851	0%	0%
PA-IDW-GW	**500 kGy**	84	1949.1	25%	49.39%
PA-IDW-GW	**1000 kGy**	101	1152.3	9.82%	70.07%
PA-IDW-GW	**2000 kGy**	52	465.6	53.57%	87.91%

In addition to analyzing the eBeam treated samples for PFOS and PFOA, the commercial laboratory also analyzed the samples for 29 different PFASs and precursors (Fig ES-7). With an increasing eBeam dose, there is a reduction in the concentrations of perfluorinated carboxylic acids (PFCA; e.g., PFBA, PFPeA, PFHXA, PFOA) as well as in the concentrations of Perfluorosulfonic acids (PFSA; e.g., PFBS, PFHXS, PFOS and PFDS).

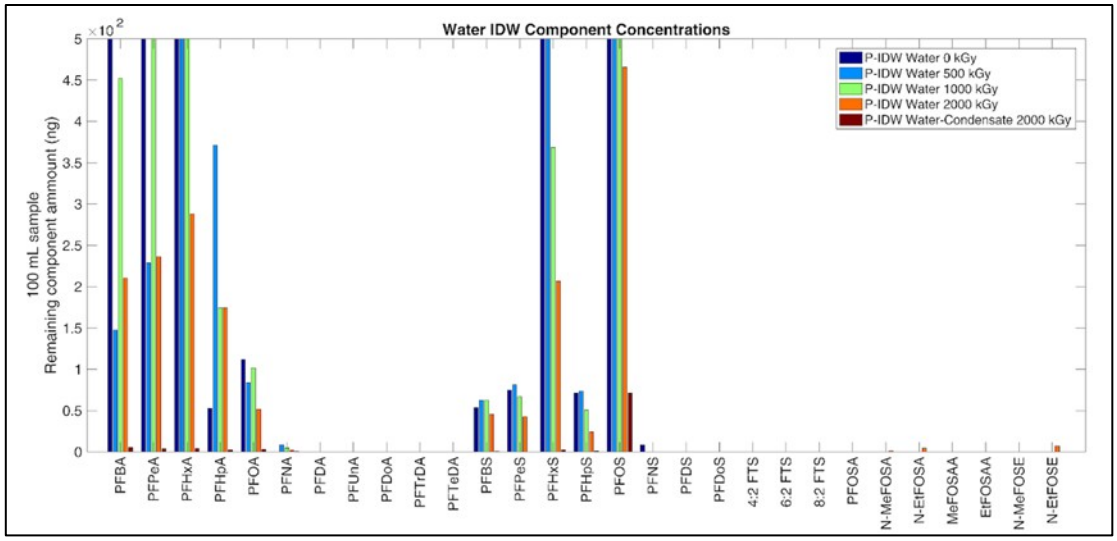
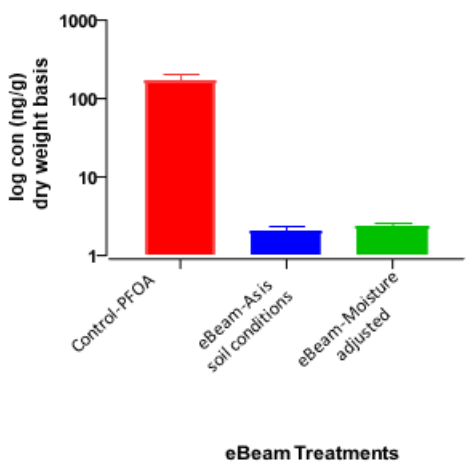


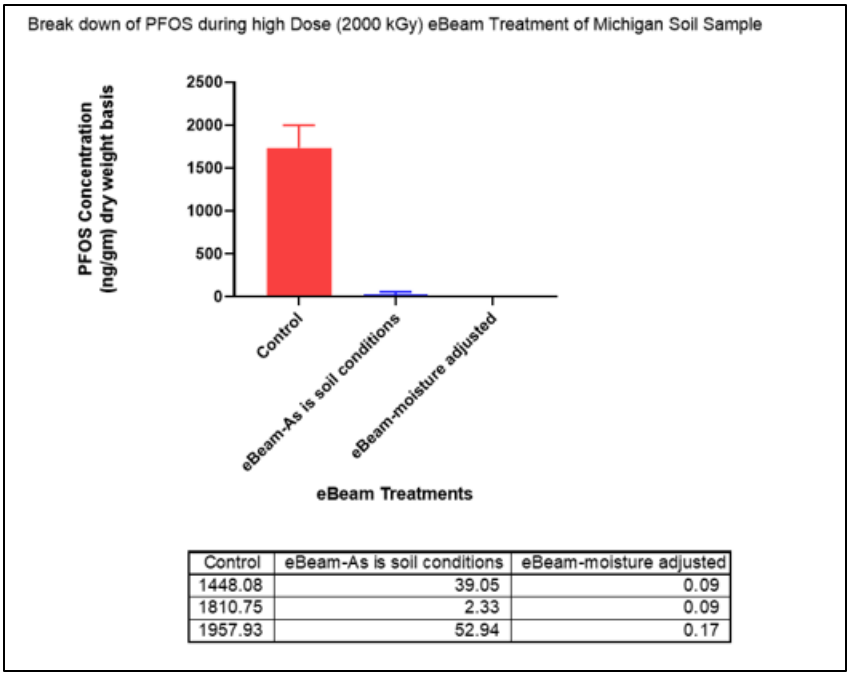
Figure ES-7. Targeted PFAS Component Concentrations in Pennsylvania IDW Water as a Function of Increasing eBeam Dose

As greater PFOS degradation in the sand compared to the aqueous samples were observed, the project team sought to confirm the hypothesis that lower moisture facilitated eBeam-mediated breakdown of PFOS in sand samples. Therefore, the project team adjusted to the moisture content of the Michigan soil sample to 10%. The project team exposed the 10% moisture-adjusted and the un-adjusted Michigan IDW soil samples to 2000 kGy eBeam dose. Figure ES-8 and Figure ES-9 show the breakdown of PFOA and PFOS in the Michigan IDW samples at 2000 kGy.



Control-PFOA	eBeam-As is soil conditions	eBeam-Mositure adjusted
129.93	2.30	2.64
206.90	1.81	2.17
172.38	2.16	2.27

Figure ES-8. Breakdown (98.7%) of PFOA During High Dose (2000 kGy) eBeam Treatment of Michigan IDW Soil Sample.



Control	eBeam-As is soil conditions	eBeam-moisture adjusted
1448.08	39.05	0.09
1810.75	2.33	0.09
1957.93	52.94	0.17

Figure ES-9. Breakdown (>99.99%) of PFOS During High Dose (2000 kGy) eBeam Treatment of Michigan IDW Soil Sample

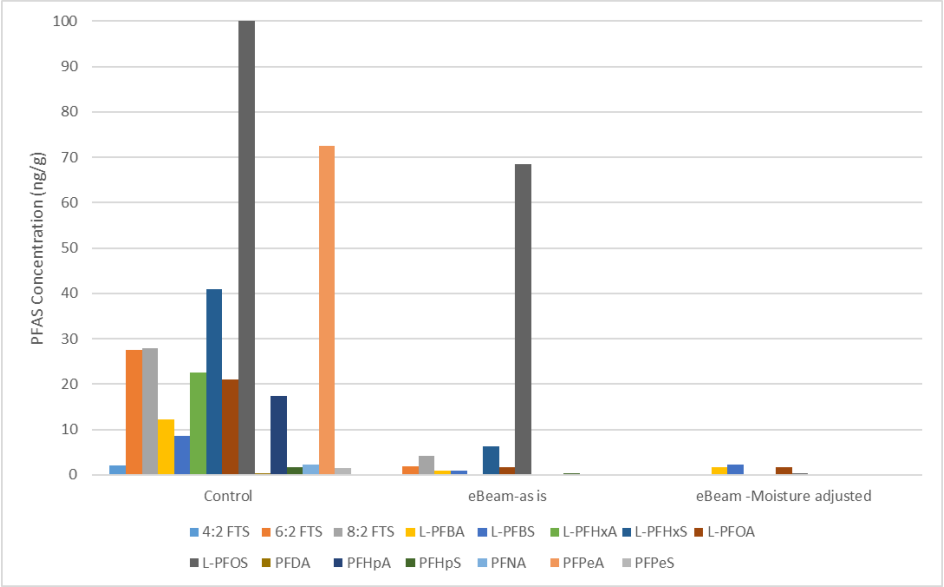


Figure ES-10. Breakdown of Targeted PFAS Components During High Dose (2000 kGy) eBeam Treatment of Michigan IDW Soil Sample

An ANOVA test was performed to compare the PFOA concentrations between the un-treated and the moisture adjusted and unadjusted treatment groups. There was a significant difference ($p<0.0001$) between the treatment groups. When a Student t-test was performed to compare the remaining PFOA concentrations in the eBeam treated samples, there was no significant difference which suggests that irrespective of whether the soil was moisture adjusted to 10% or treated as is,

PFOA can degrade by as much as 98.7%. In the case of PFOS, the breakdown was even more significant (> 99.99%) with complete degradation of PFOS (Fig. ES-9). In the IDW soil which was treated with eBeam at ambient soil moisture conditions (~ 85%), the PFOS reduction was 98.19%. In samples where the soil moisture was adjusted to 10%, the PFOS degradation was almost complete (99.99%). Figure ES-10 shows the degradation of the different targeted PFAS in the Michigan IDW samples in the 10% moisture adjusted and un-adjusted soil samples at 2000 kGy. These results suggest that PFAS degradation, especially PFOS, appears to be enhanced under low moisture conditions under high eBeam dose conditions.

Overall, the results support the original research hypothesis that eBeam can achieve significant reduction of PFOA and PFOS in IDW soil and groundwater samples.

5.0 ECONOMIC ANALYSIS

Figure ES-11 represents the cost breakdown (electrical, capital and technical personnel) for the eBeam treatment of PFAS-contaminated soils using a 10 MeV, 560 kW fixed eBeam technology platform at a 1500 kGy eBeam dose. The analysis suggests that it would cost approximately \$295/m^3 of soil for PFOS and PFOA remediation using the fixed eBeam technology platform.

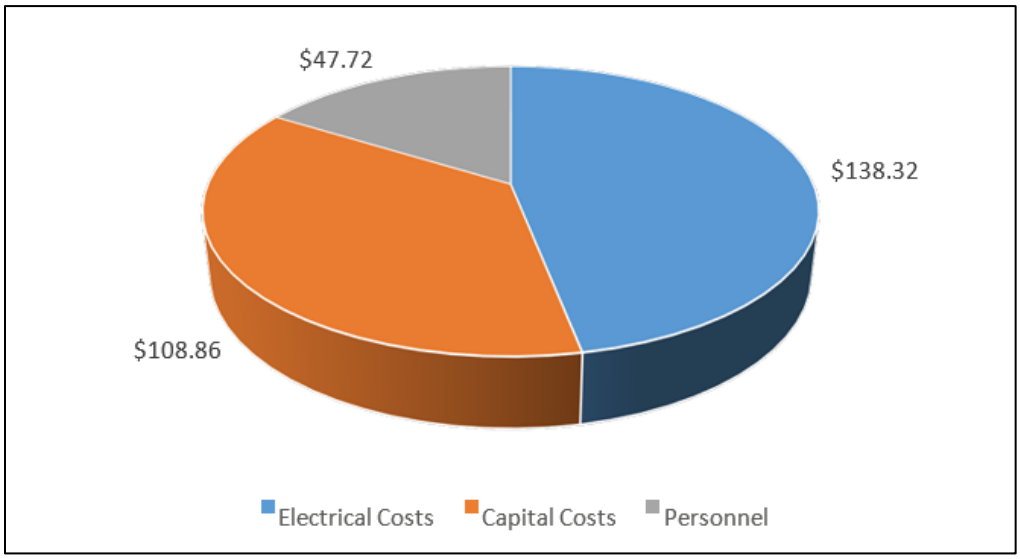


Figure ES-11. Breakdown of Costs \$/m³to treat PFAS-contaminated IDW Soils Using a Fixed Onsite eBeam Technology Platform

This analysis assumes a target dose of 1500 kGy and total capital costs of \$7.7 million depreciated over a useful life of 20 years. The assumptions include 80% beam uptime, 20 m^3/day throughput, and 90% beam utilization efficiency. Optimizing the process to achieve lower target doses and ability to utilize higher beam power will reduce the costs significantly.

The laboratory experiments support the original research hypothesis that high energy eBeam technology has potential as a suitable remediation technology for PFAS-contaminated soils and groundwater samples. To the best of the project team's knowledge, there is no technology (other than incineration) that is effective against PFOS-contaminated IDW soil and sand samples. It must

be emphasized that in this study, other than some coarse dose adjustments (between 50 kGy and 2000 kGy), eBeam technology for PFAS remediation has not been optimized. Optimization trials should include identification of the optimal pH range, the addition of appropriate amendments/radical scavengers, nitrogen sparing, calcium carbonate as well as exploration of the use of suitable catalysts such as $Ti0_2$ and $KMnO_2$. There is a need to better understand the mass balance of fluorine during eBeam remediation and the influence of presence of co-contaminants such as solvents, petroleum hydrocarbons and other PFASs on defluorination and breakdown of PFOS and PFOA. There is a need to design, fabricate, and utilize continuous flow reactors (for groundwater) and a movable conveyance system/platform to demonstrate eBeam mediated degradation of PFOS and PFOA under quasi-realistic conditions. Laboratory studies using a continuous eBeam test platform are needed to yield high value information before installing the technology onsite.

6.0 IMPLICATIONS FOR FUTURE RESEARCH AND BENEFITS

Electron beam technology is already commercially available and is employed in other applications such as medical device sterilization and food pasteurization. Thus, the Technology Readiness Level (TRL) is already very high. Electron beam technology with proper optimization and engineering design effort could become an extremely valuable tool in the Department of Defense technology "tool-box" to deal with environmental contaminants.

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