



# Six pilot-scale studies evaluating the in situ treatment of PFAS in groundwater

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## Abstract

Per- and polyfluoroalkyl substances (PFAS) have been identified by many regulatory agencies as emerging contaminants of concern in a variety of media including groundwater. Currently, there are limited technologies available to treat PFAS in groundwater with the most frequently applied approach being extraction (i.e., pump and treat). While this approach can be effective in containing PFAS plumes, previous studies of pump and treat programs have met with limited remedial success. In situ treatment studies of PFAS have been limited to laboratory and a few field studies. Six pilot-scale field studies were conducted in an unconfined sand aquifer coimpacted by petroleum hydrocarbon along with PFAS to determine if a variety of reagents could be used to attenuate dissolved phase PFAS in the presence of petroleum hydrocarbons. The six reagents consisted of two chemical oxidants, hydrogen peroxide ( $H_2O_2$ ) and sodium persulfate ( $Na_2S_2O_8$ ), and four adsorbents, powdered activated carbon (PAC), colloidal activated carbon (CAC), ion-exchange resin (IER), and biochar. The reagents were injected using direct push technology in six permeable reactive zone (PRZ) configurations. Groundwater concentrations of various PFAS entering the PRZs ranged up to 24,000  $\mu\text{g/L}$  perfluoropentanoic acid, up to 6,200  $\mu\text{g/L}$  pentafluorobenzoic acid, up to 16,100  $\mu\text{g/L}$  perfluorohexanoic acid, up to 6,080  $\mu\text{g/L}$  perfluoroheptanoic acid, up to 450  $\mu\text{g/L}$  perfluorooctanoic acid, and up to 140  $\mu\text{g/L}$  perfluorononanoic acid. Performance groundwater sampling within and downgradient of the PRZs occurred for up to 18 months using single and multilevel monitoring wells. Results of groundwater sampling indicated that the PFAS were not treated by either the persulfate nor the peroxide and, in some cases, the PFAS increased in concentration immediately following the injection of peroxide and persulfate. Concentrations of PFAS in groundwater sampled within the PAC, CAC, IER, and biochar PRZs immediately after the injection were determined to be less than the method detection limits. Analyses of groundwater samples over the 18-month monitoring period, indicated that all the PRZs exhibited partial or complete breakthrough of the PFAS over the 18-month monitoring period, except for the CAC PRZ which showed no PFAS breakthrough. Analysis of cores for the CAC, PAC, and biochar PRZs suggested that the CAC was uniformly distributed within the target injection zone, whereas the PAC and biochar showed preferential injection into a thin coarse-sand seam. Similarly, analysis of the sand packs of monitoring wells installed before the injection of the

CAC, PAC, and biochar indicated that the sand packs of the PAC and biochar preferentially accumulated the reagents compared with the reagent concentrations within the surrounding aquifer by up to 18 times.

## 1 | INTRODUCTION

The use of in situ approaches for the treatment of groundwater impacted by various contaminants of concern, including petroleum hydrocarbons and chlorinated solvents is well established (Huling & Pivetz, 2006; ITRC, 2005; Kashir & McGregor, 2015; Petri et al., 2011; Sra, Thomson, & Barker, 2013; Usman, Faure, Ruby, & Hanna, 2012). The three most frequently applied in situ methods are in situ chemical oxidation, in situ chemical reduction, and enhanced bioremediation with which each approach offering advantages over other in situ and ex situ technologies including reduced remedial time frames and costs. With the growing awareness of per- and poly-fluoroalkyl substances (PFAS) in the environment and the associated efforts to assess, contain, and remediate PFASs in a variety of media, including groundwater, a greater focus is being applied to potential technologies that can treat PFASs both ex situ and in situ (Darlington, Barth, & McKernan, 2018; ITRC, 2018; Ross et al., 2018). Simon (2020) reports that there may be greater than 42,000 PFAS-impacted sites which could cost over \$80 billion to address. The remediation of PFAS-impacted groundwater is complex due to numerous factors including that they are highly recalcitrant to biological and chemical degradation due to the strong carbon-fluoride bonding (National Ground Water Association [NGWA], 2017), low regulatory guidelines, complicated environmental fate, and limited proven remedial approaches (Simon et al., 2019).

The application of in situ technologies for the treatment of PFAS is an emerging field with only limited laboratory and field studies being conducted (Bruton & Sedlak, 2017; Carey & McGregor, 2019; Dombrowski et al., 2018; Eberle, Ball, & Boving, 2017; McGregor, 2018; Park, Lee, Medina, Zull, & Waisner, 2016). The use of in situ methods for the treatment of PFAS potentially offers several advantages over extraction technologies including higher effectiveness and lower life-cycle costs. However, there are limitations to using in situ approaches for PFAS including treatment effectiveness (Dombrowski et al., 2018), delivery and distribution of the remedial reagents, inefficient consumption of the reagents by scavenging compounds (Saeed, 2011), and matrix-diffusion and desorption issues in heterogeneous geologic materials (Carey, Chapman, Parker, & McGregor, 2015).

Testing for the ex situ treatment of PFAS-impacted water using activated carbon and ion-exchange resin (IER) have been studied extensively. However, only limited laboratory testing of reagents for the in situ treatment of PFAS have been conducted. Aly et al. (2019) demonstrated that cationic polymers mixed with soil can be effective in attenuating various PFAS in column studies, while Wang and Shih (2011) and Barajas (2019) showed that alumina and biochar can act as adsorbents for PFAS, respectively.

McGregor (2018) demonstrated that colloidal activated carbon (CAC) could be injected and attenuate select PFAS in a dilute groundwater plume at a site in Central Canada situated in an unconfined silt and sand aquifer. Based on these results and laboratory tests completed with a variety of chemical oxidants, including persulfate and hydrogen peroxide, and adsorptive media, including powdered activated carbon (PAC), CAC, biochar and IER, a series of pilot tests were conducted to evaluate the effectiveness of each reagent in a permeable reactive zone (PRZ) configuration.

### 1.1 | Site overview

The site is underlain by an unconfined aquifer consisting of fine-grained sand with a 0.04-m thick medium sand seam located at a depth of 6.2 m below ground surface (mbgs). The water table occurs at a depth of ~5.3 m below grade. In addition to being impacted with PFAS, the shallow aquifer was also impacted with petroleum hydrocarbons in the gasoline range with concentrations up to 3,500 ug/L being measured in the groundwater flowing into the PRZs.

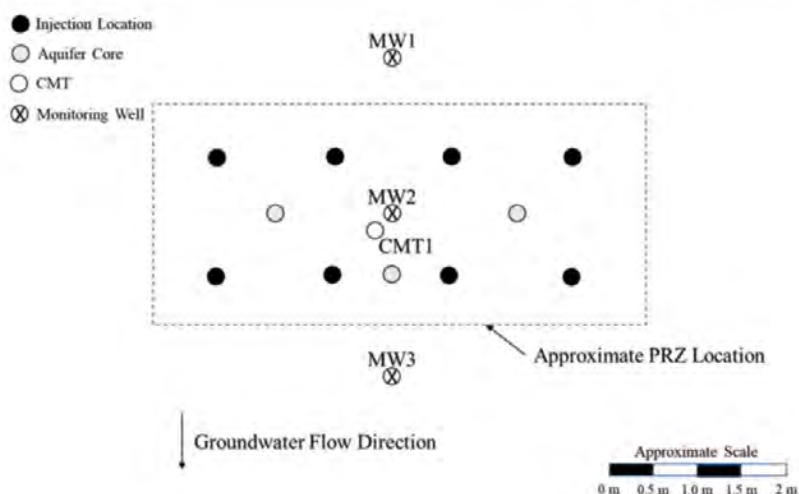
Each pilot-test area covered ~40 m<sup>2</sup> and was instrumented with three monitoring wells (MW1–MW3) and one 3-channel multitubing (CMT) well (CMT1). Each polyvinyl chloride monitoring well was constructed with a 1.5-m long screen situated across the water table surrounded by a 0.2-m diameter coarse-grained sand pack overlain by a bentonite seal. Figure 1 provides a general schematic of the layout of each test area.

## 2 | METHODOLOGY

The six reagents were injected using direct push technology (DPT) at eight locations in each test cell to form the six separate segments of PRZ. The six reagents injected were: PAC, CAC, IER, biochar, hydrogen peroxide, and sodium persulfate. A Geoprobe™ 7822 equipped with 0.038-m diameter hollow rods and a 0.3-m long screened injection tool was used to inject the reagent solutions on a 1.5-m grid (Figure 1). The six reagents were injected at a 10% by weight solution at pressures ranging from 20 to 250 pounds per square inch. The reagents were injected using a bottom-up approach based on 0.3-m vertical intervals. The volume of reagent solution was determined by targeting 40% of the effective pore volume of the PRZ based on an effective porosity of 0.2.

The reagents used in this study were purchased from a variety of suppliers and included the CAC which was purchased from Regenesis® (San Clemente, CA) and is sold under the brand name PlumeStop™ and had a mean diameter of 1–2 μm and a specific

**FIGURE 1** Generalized layout of each permeable reactive zone (PRZ) showing monitoring and CMT wells along with injection and coring locations. CMT, channel multitubing; MW, monitoring well [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



gravity of up to 1.2. The PAC injected was Calgon Carbon Corporation (Moon Township, PA) WPC™ which had a mean grain size of ~100  $\mu\text{m}$ . The IER used in the study was a single use anion exchange resin sold under the brand name Purolite® (Bala Cynwyd, PA) Pur-ofine® PFA694E™ with a mean diameter of the IER is 675  $\mu\text{m}$  ( $\pm 75 \mu\text{m}$ ) and specific gravity of 1.05. The biochar was obtained from a local supplier and had a mean grain size of 180  $\mu\text{m}$ . The sodium persulfate was purchased from PeroxyChem (Philadelphia, PA) and is sold under the brand name Klozur™ whereas the hydrogen peroxide was purchased from a local distributor.

To evaluate the effectiveness of the reagents for the removal of the PFAS, groundwater samples were collected on seven occasions from monitoring well MW2 within each test area; twice before injection of the reagents to establish a baseline concentration and five times postinjection on Days 92, 184, 278, 366, and 549. The groundwater samples were collected using low-flow sampling methods (Puls & Barcelona, 1996). Selected field parameters were measured at the wellhead using a flow-through cell connected to a YSI Professional Plus hand-held multiparameter meter (YSI, Inc., Yellow Springs, OH). The parameters measured at the wellhead included pH, oxidation–reduction potential (ORP), temperature, specific conductivity, turbidity, and dissolved oxygen (DO). The pH meter was calibrated to solutions of 4.0, 7.0, and 10.0 standard units before each sampling event. A calibration check was performed before collecting each sample. The ORP was checked with Zobell's solution (Nordstrom, 1977) before each sample measurement and electrical conductivity was calibrated to a National Institute of Standards and Technology traceable standard solution.

Groundwater samples were collected for analysis of a variety of inorganic and PFAS compounds during the pilot-test program. A total of 23 PFAS compounds consisting of the following were analyzed:

- 6:2 Fluorotelomer sulfonic acid (6:2),
- 8:2 fluorotelomer sulfonic acid (8:2),
- N-ethyl perfluorooctane sulfonamide,

- N-ethyl perfluorooctane sulfonamidoethanol,
- N-methyl perfluorooctane sulfonamide,
- N-methyl perfluorooctane sulfamidoethanol,
- perfluorobutanesulfonic acid,
- pentafluorobenzoic acid (PFBA),
- perfluorodecanesulfonate,
- perfluorodecanoic acid,
- perfluorododecane sulfonic acid,
- perfluorododecanoic acid,
- perfluorooctane sulfonamide,
- perfluorohexanoic acid (PFHxA),
- perfluoroheptanoic acid (PFHpA),
- perfluorohexane sulfonate,
- perfluorononanoic acid (PFNA),
- perfluorooctanesulfonic acid,
- perfluorooctanoic acid (PFOA),
- perfluoropentanoic acid (PFPeA),
- perfluorotetradecanoic acid,
- perfluorotridecanoic acid, and
- perfluoroundecanoic acid.

The samples were collected using laboratory-supplied high-density polyethylene containers and shipped to a commercial laboratory for analyses using EPA Method 533, 537, and 537.1.

Inorganic parameters, including chloride ( $\text{Cl}^-$ ), sulfate ( $\text{SO}_4^{2-}$ ), nitrate ( $\text{NO}_3^-$ ), dissolved iron (Fe), dissolved manganese (Mn), and alkalinity, were also analyzed by a commercial laboratory. The samples for  $\text{SO}_4$ ,  $\text{NO}_3$ , and Cl were not filtered, whereas the samples for alkalinity, dissolved Fe, and dissolved Mn were filtered with a 0.45  $\mu\text{m}$  cellulose acetate membrane. The samples for dissolved Fe and Mn were acidified to pH below 2 with nitric acid ( $\text{HNO}_3$ ). Groundwater samples were also collected for total organic carbon (TOC) and analyzed using a Shimadzu Carbon Analyzer following EPA Method 415.1.

Horizontal hydraulic conductivity ( $K_H$ ) estimates were completed using a flexible wall permeameter (ASTM International, Inc., 2016, [ASTM] D5084-16a) by collecting continuous cores of the aquifer and subdividing the cores in 0.2 m-long samples. Cores were collected prior and postinjection to evaluate any effects of the reagents on the  $K_H$  of the aquifer. The tests were completed under constant-head conditions using site groundwater. Before testing for  $K_H$ , samples of each core were collected for analysis of the preinjection TOC to determine the organic matter content of the aquifer before the injection.

Additional cores of the aquifer were collected from within the PRZs 1 day postinjection to observe the distribution of the reagents as well as to determine the TOC content within the pre-existing monitoring well sand packs. Continuous cores for the PAC, CAC, and biochar PRZs were sampled and submitted to a commercial laboratory for TOC following the combustion methods outlined by the ASTM (2011, 2013). Cores from the  $\text{Na}_2\text{S}_2\text{O}_8$  and  $\text{H}_2\text{O}_2$  PRZs were collected and pore water was extracted using a combination of immiscible fluid, displacement, suction, and mechanical compression (Moncur, Blowes, & Ptacek, 2013). The pore water was then analyzed using Chemetric™ test kits for persulfate and hydrogen peroxide to estimate the concentration of persulfate and hydrogen peroxide. No cores for the IER PRZ were collected and analyzed due to analytical challenges associated with the analysis of the IER material.

### 3 | RESULTS AND DISCUSSION

#### 3.1 | Horizontal hydraulic conductivity

The effectiveness of any in situ treatment is limited by the effectiveness of the delivery of the reagents being injected which in turn is limited by the  $K_H$  of the aquifer  $K_H$  the reagent is being injected into. In general, the delivery and distribution of the reagents will be more

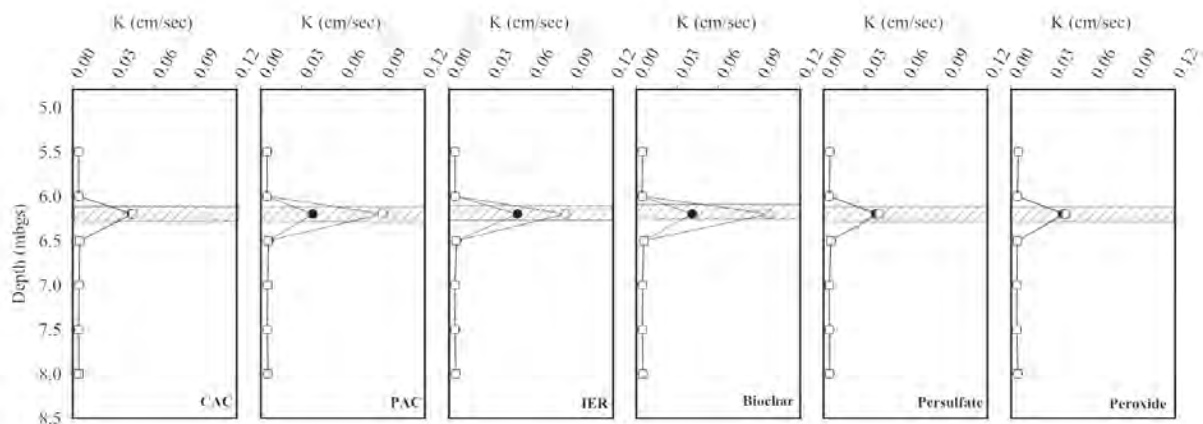
effective in higher hydraulic conductivity zones and, thus, more reagent will be delivered to these zones compared with lower  $K_H$  zones. A series of continuous cores of the aquifer were collected for each PRZ before and postinjection to evaluate the vertical variability of the  $K_H$  as well as the possible effect of the reagents on the  $K_H$ .

The results of the  $K_H$  testing indicated that much of the impacted aquifer (i.e., fine sand) had a  $K_H$  ranging from  $4.1 \times 10^{-5}$  m/s to  $5.7 \times 10^{-5}$  m/s, averaging  $4.7 \times 10^{-5}$  m/s ( $n = 36$ ). Measurements of the  $K_H$  of the coarser sand seam ranged from  $3.8 \times 10^{-4}$  to  $5.0 \times 10^{-4}$  m/s averaging  $4.7 \times 10^{-4}$  m/s ( $n = 6$ ), representing an order of magnitude difference in the  $K_H$  between the coarser sand seam and the surrounding fine-grain sand aquifer. Figure 2 provides vertical profiles of the  $K_H$  for each PRZ.

#### 3.2 | Hydrogeology

Horizontal hydraulic gradients averaged 0.008 m/m during the testing period. Using this horizontal hydraulic gradient and an average  $K_H$  of  $4.7 \times 10^{-5}$  m/s, the mean horizontal groundwater velocity of the fine-grained sand aquifer was estimated to be approximately ~6 m/year based on an assumed effective porosity of 0.2. Instrumentation of the coarse-grained sand seam was completed using CMTs with 0.15 m long screens. Using a horizontal hydraulic gradient of 0.008 m/m and an average  $K_H$  of  $4.7 \times 10^{-4}$  m/s and an effective porosity of 0.2, an average groundwater velocity of ~60 m/year was estimated, indicating that the groundwater flux through the coarse sand seam was ~10 times greater than the surrounding finer-grained sand.

Based on these estimated velocities for the fine-grained aquifer, it is estimated that the PRZs had experienced 0.6, 1.2, 1.8, 2.4, and 3.6 pore volumes of flow on Days 92, 184, 278, 366, and 549, respectively. Using the estimated groundwater velocity for the coarse-grained sand seam, it is estimated that 6.0, 12, 18, 24, and



**FIGURE 2** Horizontal hydraulic conductivity ( $K_H$ ) profiles for PRZs. The black circles (●) represent the preinjection  $K_H$  whereas the white squares (□) represent postinjection  $K_H$  values. The hatched area represents the coarse-grained sand seam. CAC, colloidal activated carbon; IER, ion-exchange resin; PAC, powdered activated carbon; PRZs, permeable reactive zones

36 pore volumes of flow had passed through the PRZs on Days 92, 184, 278, 366, and 549, respectively.

The preinjection TOC of the aquifer was determined by collecting a continuous core within each PRZ before the injection of the reagents. A total of 48 samples were analyzed with the mean preinjection TOC being 0.0061 g/kg ( $n = 48$ , standard deviation [SD] = 0.0009 g/kg; Figure 3). The preinjection TOC within the target injection interval between 5.5 and 7.0 mbgs was 0.0062 g/kg ( $n = 30$ ,  $SD = 0.0009$ ) suggesting that the preinjection TOC concentration was relatively uniform throughout the vertical profiles of each core.

### 3.3 | Reagent distribution

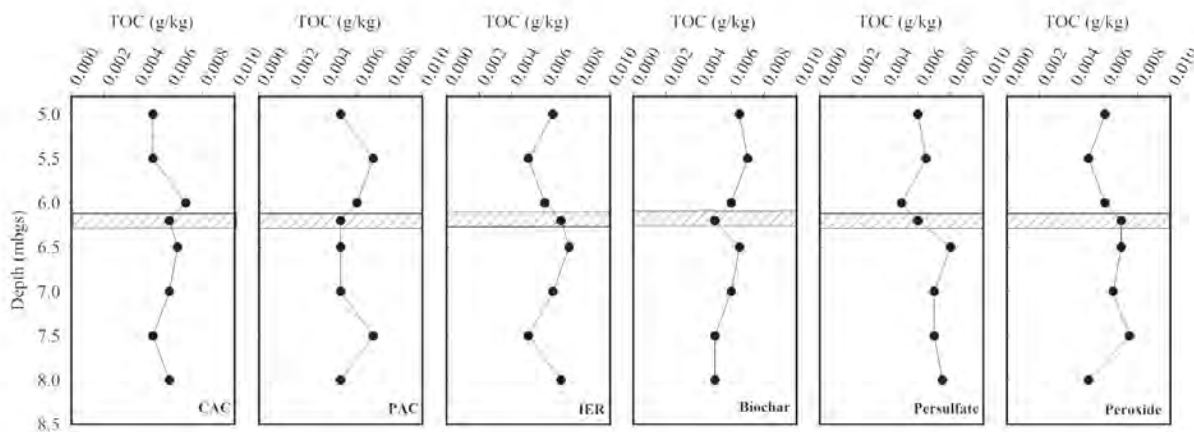
A key factor in achieving effective in situ treatment is the uniform distribution of the reagents within the target zone. Due to the relatively low groundwater velocity at the study site, the reagents are primarily distributed during the injection of the solution and not by subsequent advection/dispersion processes. Advection and dispersion can aid in the distribution of some of the persistent and fine-grained reagents such as CAC and persulfate; however, these processes are limited and were not relied on in the design of the PRZs to distribute the reagents at the study site.

To evaluate the distribution of the reagents, three continuous soil cores were collected within each PRZ over the depth of the targeted injection zone within aquifer (Figure 1). The two reagents that showed the most uniform distribution over the targeted injection zone were CAC and persulfate (Figures 4 and 5). Analysis of the 27 samples collected for TOC within the CAC PRZ indicated that the mean TOC concentration within the targeted injection zone was 0.77 g/kg ( $n = 15$ ,  $SD = 0.096$  g/kg) compared with a mean TOC concentration of 0.11 g/kg ( $n = 12$ ,  $SD = 0.029$  g/kg) outside of the targeted injection zone (Figure 4). Before the injection of the CAC, the preinjection TOC of the soil over the same interval was 0.0056 g/kg

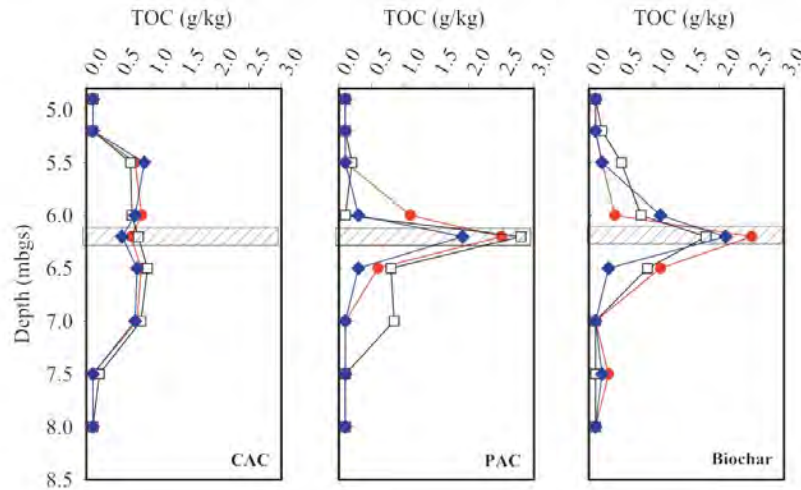
( $n = 8$ ,  $SD = 0.0009$  g/L; Figure 3). These results indicate that the TOC within the injection zone was seven times greater than outside the injection zone and approximately two orders of magnitude greater than the preinjection TOC of the soil before the injection. All 15 samples collected within the targeted injection zone had detectable concentrations of TOC compared with 33% of the samples collected outside of the targeted injection zone (Figure 4).

Analysis of pore-water samples collected for persulfate within the persulfate PRZ indicated that the mean persulfate concentration within pore water of the targeted injection zone was 51.6 g/L ( $n = 15$ ,  $SD = 22.9$  g/L) compared with a mean persulfate concentration of 0.21 g/L ( $n = 12$ ,  $SD = 2.63$  g/kg) outside of the targeted injection zone (Figure 5). The results indicate that the persulfate within the injection zone was 4.5 orders of magnitude greater than the pore water persulfate concentration outside the injection zone. All pore water samples collected within the targeted injection zone had detectable persulfate 24 hr post-injection ( $n = 15$ ). Outside of the target injection zone, 25% of the pore water samples had detectable persulfate suggesting that most of the persulfate solution was injected into the target zone (Figure 5).

Analysis of the pore water and aquifer solids for the PAC, biochar, and peroxide PRZs showed significant variability in the distribution of the reagents within the targeted injection zone. Analysis of the TOC within the PAC and biochar PRZs indicated an enrichment of TOC within the coarse-grained sand seam located at a depth of 6.2 mbgs (Figure 4). The concentration of TOC within the sand seam in the PAC PRZ averaged 2.40 g/kg ( $n = 3$ ) compared with the TOC average of 0.79 g/kg for the targeted injection zone ( $n = 15$ ,  $SD = 0.908$  g/kg; Figure 4). Similarly, the concentration of TOC within the sand seam in the biochar PRZ averaged 2.13 g/kg ( $n = 3$ ) compared with the TOC average of 0.81 g/kg for the targeted injection zone ( $n = 15$ ,  $SD = 0.777$  g/kg; Figure 4). Analysis of the samples for TOC within the PAC and biochar targeted injection zones indicated that 67% and 75% of the samples had detectable TOC concentrations, respectively (Figure 4).



**FIGURE 3** Preinjection TOC profiles for aquifer solids collected within each of the PRZs. The hatched area represents the coarse-grained sand seam. CAC, colloidal activated carbon; IER, ion-exchange resin; PAC, powdered activated carbon; PRZs, permeable reactive zones; TOC, total organic carbon



**FIGURE 4** Concentration profiles for TOC in aquifer solids for cores collected from CAC, PAC, and Biochar PRZs. Red circles (●) indicate results from Core 1, white squares (□) represent results from Core 2 and blue diamonds (◆) represent results from Core 3. The hatched area represents the coarse-grained sand seam. CAC, colloidal activated carbon; PAC, powdered activated carbon; PRZs, permeable reactive zones; TOC, total organic carbon [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

Analysis of the TOC within the soil outside of the targeted injection zones for the PAC and biochar PRZs indicated that the majority of the reagents were injected into the targeted injection zone with 17% and 50% of the samples outside of the targeted injection zone having detectable concentrations of TOC, respectively (Figure 4).

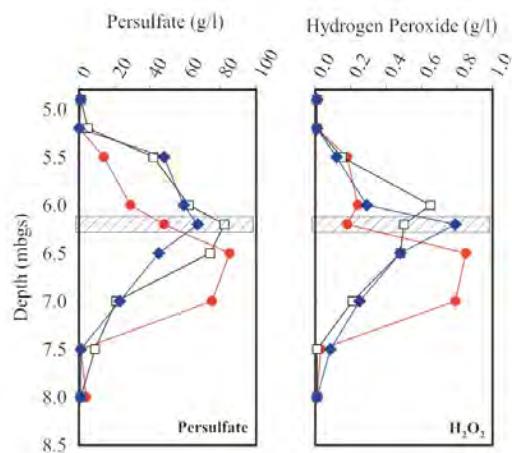
Analysis of pore-water samples collected for  $H_2O_2$  within the peroxide PRZ indicated that the mean  $H_2O_2$  concentration within pore water of the targeted injection zone was 0.41 g/L ( $n = 15$ ,  $SD = 0.257$  g/L) compared with a mean  $H_2O_2$  concentration of 0.02 g/L ( $n = 12$ ,  $SD = 0.022$  g/L) outside of the targeted injection zone (Figure 5). The results indicate that the  $H_2O_2$  within the injection zone was ~1,950% greater than the pore water  $H_2O_2$  concentration

outside the injection zone 24 hr after injection. All pore water samples collected within the targeted injection zone had detectable  $H_2O_2$  24 hr postinjection ( $n = 15$ ), whereas, 17% of the pore water samples outside of the target injection zone had detectable  $H_2O_2$  suggesting that the majority of  $H_2O_2$  solution was injected into the target zone (Figure 5).

### 3.4 | Well screens

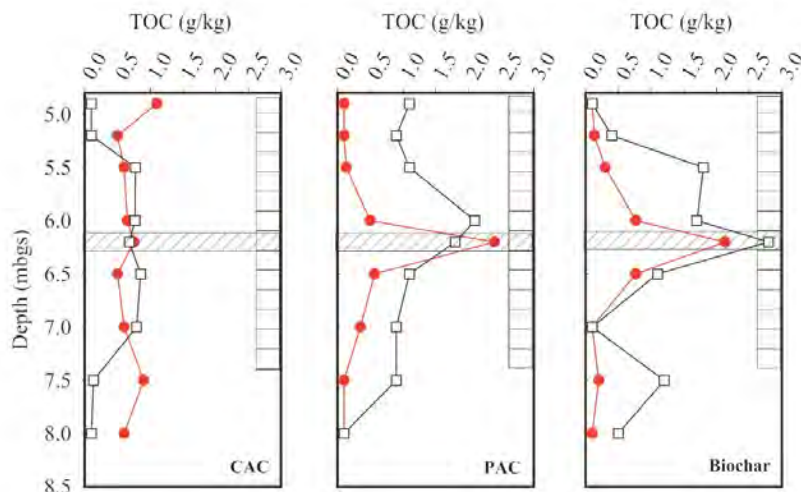
Studies by Fan, Gilbert, and Fox (2017) indicated that there could be preferential accumulation of powdered and granular activated carbon within pre-existing monitoring well sand packs following the injection of PAC. This preferential accumulation of activated carbon, and other solid-based reagents, could result in nonrepresentative groundwater samples being collected and used for the evaluation of the effectiveness of remedial activities. To evaluate if this preferential accumulation was occurring within the test sites, continuous cores of the sand packs of monitoring wells were collected within the CAC, PAC, and biochar PRZs before the injection of the reagents. The results of the analysis of the cores for TOC suggest that preferential accumulation occurred within the sand packs during the injection of the coarser-grained reagents including PAC and biochar. The TOC concentrations for the sand pack within the PAC PRZ ranged from 0.90 to 2.10 g/kg, averaging 1.40 g/kg, compared with a range of 0.10–2.80 g/kg and a mean of 0.79 g/kg for the surrounding targeted injection zone (Figure 6). The concentrations of TOC for the biochar PRZ monitoring well sand pack ranged from 0.10 to 2.80 g/kg, averaging 1.50 g/kg compared with a range of 0.10–2.50 g/kg and a mean of 0.81 g/kg for the surrounding targeted injection zone (Figure 6).

The results of the TOC analysis for the sand pack within the CAC PRZ showed that the TOC concentration of the sand pack ranged from 0.50 to 0.75 g/kg, averaging 0.62 g/kg compared with a range of 0.55–0.80 g/kg and a mean of 0.77 g/kg for the surrounding targeted



**FIGURE 5** Concentration profiles for dissolved persulfate and hydrogen peroxide in pore water collected from persulfate and peroxide PRZs. Red circles (●) indicate results from Core 1, white squares (□) represent results from Core 2 and blue diamonds (◆) represent results from Core 3. The hatched area represents the coarse-grained sand seam. PRZs, permeable reactive zones [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

**FIGURE 6** Total organic carbon (TOC) profiles for sand packs within CAC, PAC, and biochar PRZs. Square symbols ( $\square$ ) represent TOC concentrations within sand pack and red circles ( $\bullet$ ) represent mean TOC concentrations in surrounding aquifer. Diagonal hashes represent the coarse-grained sand seam while the horizontal hashes represent the sand pack. CAC, colloidal activated carbon; PAC, powdered activated carbon; PRZs, permeable reactive zones [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



injection zone (Figure 6). These results suggest that the monitoring well sand pack for the CAC PRZ was not preferentially accumulating CAC for this study.

### 3.5 | General groundwater geochemistry

Groundwater samples were analyzed for select inorganic parameters to gather information on the geochemical environment and processes occurring within the pilot-test areas before and postinjection.

The mean pH of the groundwater within the five PRZs before the injection of the reagents was 7.09 ( $n = 12$ ). Six months postinjection the pH ranged from 5.89 within the persulfate PRZ to 7.93 within the PAC PRZ. The depressed pH within the persulfate PRZ is attributed to the low pH of the injected sodium persulfate solution. The mean alkalinity of the groundwater before injecting the persulfate was 428 mg/L (as  $\text{CaCO}_3$ ) whereas the alkalinity decreased to 27 mg/L (as  $\text{CaCO}_3$ ) 184 days postinjection. This decrease suggests that the acidic persulfate solution had nearly overcome the carbonate buffering capacity of the aquifer during and immediately postinjection. The pH of the groundwater on Day 366 was measured to be 6.89 suggesting that the persulfate solution was consumed by Day 366. The elevated pH of the groundwater sampled from within the PAC PRZ is likely associated with an increase in pH due to the PAC itself. It has been observed in aboveground activated carbon-based treatment systems that the pH can temporarily increase when new activated carbon is introduced into the system depending on what the activated carbon was rinsed with during preparation of the activated carbon.

The mean ORP of the groundwater before the injection of the six reagents ranged from  $-285$  to  $-98$  mV, averaging  $-161$  mV ( $n = 12$ ). The mean ORP of the groundwater sampled on Day 184 increased to  $-118$  mV ( $n = 12$ ) ranging from  $-177$  mV for the groundwater sampled within the biochar PRZ to  $-10$  mV for the groundwater sampled from the persulfate PRZ. The overall increase in ORP is mostly due to the

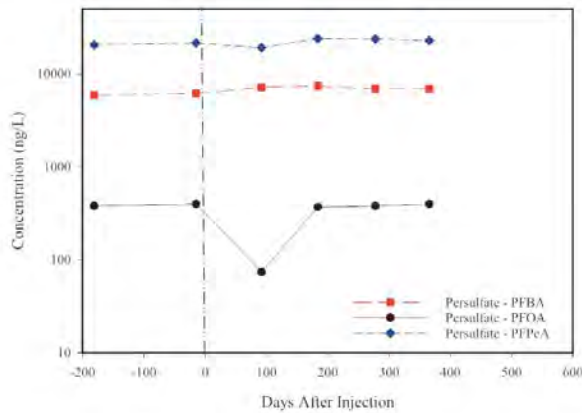
increase in ORP associated with the persulfate PRZ during the first 278 days of the test period. The ORP within the persulfate PRZ returned to within the background range after 12 months ( $-230$  mV) while the ORP measured in the groundwater of the other PRZs remained relatively constant over the test period.

DO levels within the groundwater before the injection of the six reagents ranged from 0.8 to 1.1 mg/L, averaging 0.80 mg/L ( $n = 12$ ). Following the injection of persulfate, the mean DO was 0.32 mg/L ( $n = 6$ ), indicating that oxygen content decreased over the first 184 days of the period between injection and sampling. The reason for this decrease is unclear but may be related to increased biological activity associated with the injection of some of the reagents, such as the CAC, which have an organic-based carrier solution associated with them or release oxygen over the short term thus potentially stimulating aerobic biological activity (i.e., hydrogen peroxide).

Nitrate concentrations within the groundwater before and postinjection of the six reagents remained nearly constant averaging 0.97 mg/L before the injection of the reagents versus 1.09 mg/L 184 days postinjection. The groundwater sampled from within the IER PRZ showed the greatest change in nitrate concentrations, increasing to 1.5 mg/L from 0.088 mg/L during the Day 184 sampling event. Sulfate concentrations before the injection of the six reagents averaged 2.77 mg/L, ranging from 0.43 mg/L in the groundwater sampled from the biochar PRZ to 6.0 mg/L in the CAC PRZ. On Day 184, sulfate concentrations within the groundwater sampled from the six PRZs, ranged from 1.2 mg/L in the IER PRZ to 1.6 mg/L in groundwater sampled from the PAC PRZ, averaging 1.90 mg/L.

### 3.6 | PFAS treatment

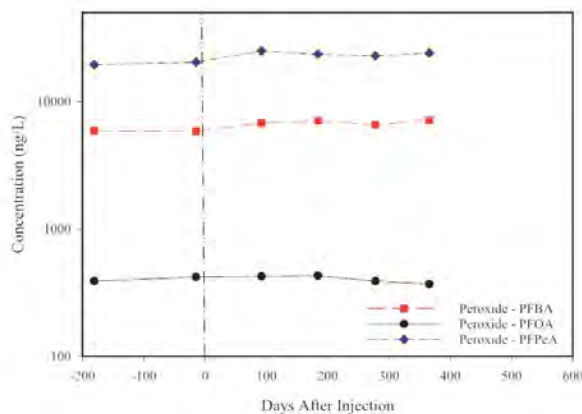
Analysis of a suite of PFAS detected six compounds at concentrations greater than their respective detection limits. The six compounds were PFBA, PFPeA, PFHxA, PFHpA, PFOA, and PFNA which have between five (PFPeA) and nine (PFNA) carbon atoms. Groundwater



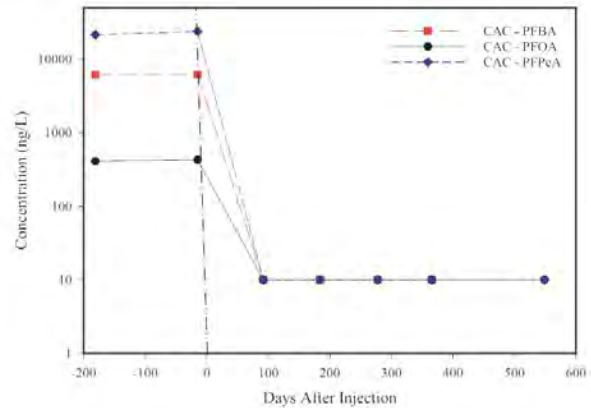
**FIGURE 7** Select PFAS concentrations with time for groundwater sampled within persulfate PRZ. The hatched vertical line indicates time of injection. PFAS, polyfluoroalkyl substances, PFBA, pentafluorobenzoic acid; PFOA, perfluorooctanoic acid; PFPeA, perfluoropentanoic acid; PRZ, permeable reactive zone [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

concentrations for the six PFAS entering the PRZs ranged up to 24,000  $\mu\text{g/L}$  for PFPeA, up to 6,200  $\mu\text{g/L}$  for PFBA, up to 16,100  $\mu\text{g/L}$  for PFHxA, up to 6,080  $\mu\text{g/L}$  for PFHpA, up to 450  $\mu\text{g/L}$  for PFOA, and up to 140  $\mu\text{g/L}$  for PFNA.

The treatment of the six PFAS using the chemical oxidants, sodium persulfate, and hydrogen peroxide, was ineffective with little to no removal of the PFAS being observed after 366 days of treatment (Figures 7 and 8). Groundwater sampled on Day 92 from within the peroxide PRZ showed no reduction of the six PFAS concentrations, with four of the PFAS (PFPeA, PFHpA, PFNA, PFBA, and PFHxA) concentrations increasing by >10% relative to their concentrations entering the PRZ. These results suggest that the hydrogen peroxide may have oxidized other potential PFAS (precursors) resulting in the



**FIGURE 8** Select PFAS concentrations with time for groundwater sampled within peroxide PRZ. The hatched vertical line indicates time of injection. PFAS, polyfluoroalkyl substances, PFBA, pentafluorobenzoic acid; PFOA, perfluorooctanoic acid; PFPeA, perfluoropentanoic acid; PRZ, permeable reactive zone [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



**FIGURE 9** Select PFAS concentrations with time for groundwater sampled within CAC PRZ. The hatched vertical line indicates time of injection. CAC, colloidal activated carbon; PFAS, polyfluoroalkyl substances; PFBA, pentafluorobenzoic acid; PFOA, perfluorooctanoic acid; PFPeA, perfluoropentanoic acid; PRZ, permeable reactive zone [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

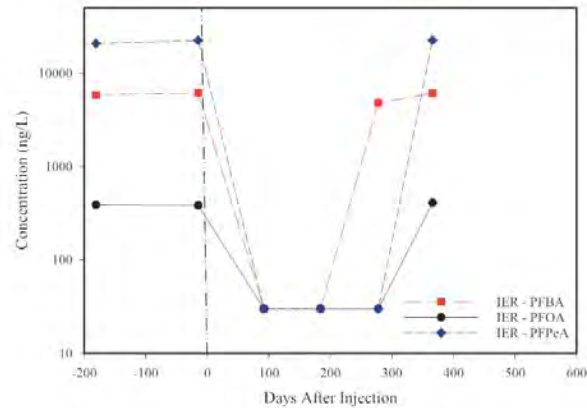
generation of the PFAS being analyzed (perfluorinated species). This process is the basis for the total oxidizable precursors analysis, which is used to estimate the PFAS precursors present within water (Houtz, Higgins, Field, & Sedlak, 2013). After 366 days of treatment, groundwater samples indicated that PFHpA, PFOA, PFHxA, and PFNA had decreased or were similar in concentration to their pre-injection concentrations, compared with increases for PFBA and PFPeA, within the peroxide PRZ (Figures 7 and 8). Similar results were observed for the Persulfate PRZ with PFBA and PFNA having an increase in concentration relative to the influent concentration within the groundwater sampled on Day 92 (Figure 7). Concentrations of the six PFAS sampled from both the peroxide and persulfate PRZs after 366 days indicated that the PFAS concentrations increased or remained the same over the testing period with the exception of PFHpA and PFOA in the groundwater sampled from the peroxide PRZ which decreased by 14% or less compared to their Day 0 concentrations (Figure 8).

Treatment of the six PFAS detected in the groundwater entering the four adsorption based PRZs (CAC, PCA, biochar, and IER) were variable with time and space, as described below.

### 3.6.1 | CAC permeable reactive zone

The concentrations of the six PFAS within the groundwater sampled from the monitoring well and CMT within the CAC PRZ were all below the detection limit during the five postinjection sampling events including samples collected on Day 549 (Figure 9). These results indicate that the PFAS were being effectively adsorbed by the CAC. Sampling and analysis of the CMT multilevel monitoring well indicated that the six PFAS were below their detection limits throughout the vertical extent of the plume including within the coarse-grained sand seam (Figure 13).





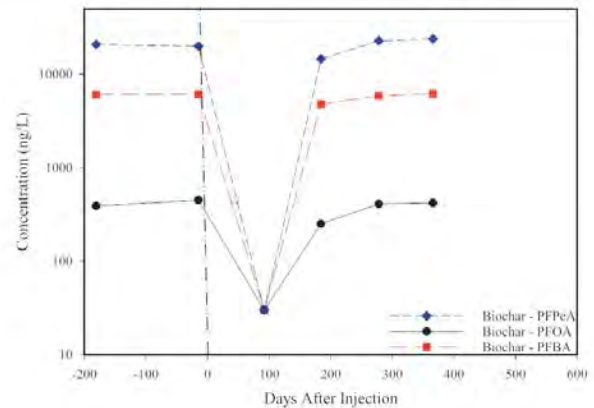
**FIGURE 10** Select PFAS concentrations with time for groundwater sampled within IER PRZ. The hatched vertical line indicates time of injection. IER, ion-exchange resin; PFAS, polyfluoroalkyl substances; PFBA, pentafluorobenzoic acid; PFOA, perfluorooctanoic acid; PFPeA, perfluoropentanoic acid; PRZ, permeable reactive zone [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

### 3.6.2 | IER permeable reactive zone

Groundwater sampled on Day 92 from the monitoring well within the IER PRZ indicated that the six PFAS had been effectively removed from solution to below the respective detection limits (Figure 10). On Day 278, PFPeA and PFHxA were detected within the groundwater with PFPeA being measured at a concentration near its influent concentration suggesting that PFPeA had broken through the IER. The PFHxA concentration was approximately ~10% of the influent concentration suggesting that PFHxA was starting to break through the IER (Figure 10). PFPeA and PFHxA represent the two shortest carbon chained PFAS detected within the groundwater with carbon chains of five and six, respectively. After 366 days of treatment, four of the six PFAS, showed a decrease in concentration whereas two, PFPeA and PFOA, showed no treatment after 366 days (Figures 10 and 13). Vertical sampling of the PFAS plume within the IER PRZ on Day 366 indicated higher concentrations of the six PFAS detected within the groundwater were detected within the coarse-grained sand seam with breakthrough occurring within the sand seam before the surrounding finer-grained sand (Figure 14).

### 3.6.3 | Biochar permeable reactive zone

Groundwater sampled on Day 92 within the biochar PRZ indicated that the six PFAS had been attenuated to below their respective detection limits (Figure 11). On Day 184, PFBA, PFHxA, PFHpA, and PFOA were detected within the groundwater indicating that these four PFAS had partially broken through the biochar (Figure 11). As with the IER, the shorter carbon chain PFAS broke through before the longer carbon chain PFAS suggesting that preferential and/or competitive adsorption may be occurring on the biochar for the PFAS. After 366 days of

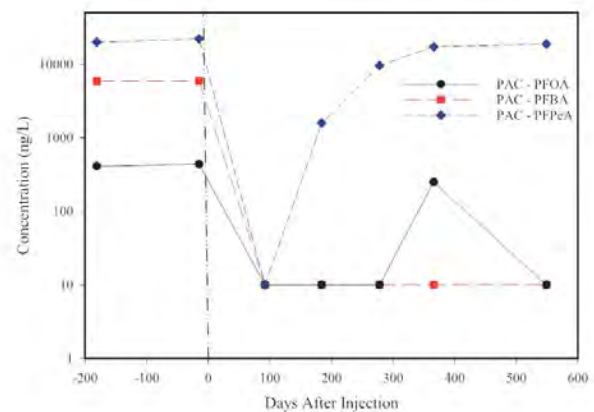


**FIGURE 11** Select PFAS concentrations with time for groundwater sampled within biochar PRZ. The hatched vertical line indicates time of injection. PFAS, polyfluoroalkyl substances; PFBA, pentafluorobenzoic acid; PFOA, perfluorooctanoic acid; PFPeA, perfluoropentanoic acid; PRZ, permeable reactive zone [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

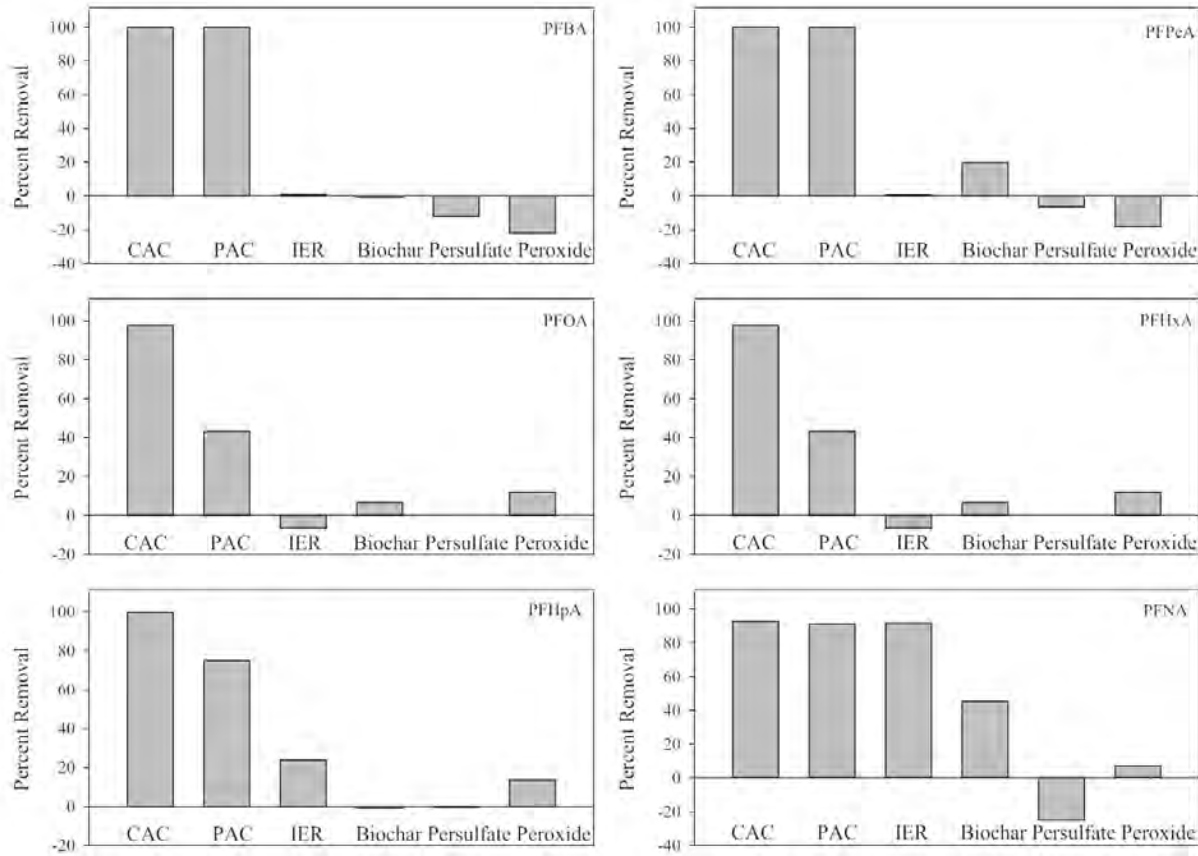
treatment, all six of the PFAS were detected within the groundwater sampled within the biochar PRZ at or near the influent concentrations indicating that the biochar attenuation capacity had been exhausted (Figures 11 and 13). As with the IER PRZ, vertical sampling of the PFAS plume within the biochar PRZ on Day 366 indicated higher concentrations of the PFAS within the coarse-grained sand seam compared with the surrounding finer-grained sand (Figure 14).

### 3.6.4 | PAC permeable reactive zone

Sampling of the groundwater within the PAC PRZ on Day 92 indicated that the six PFAS had been removed from the



**FIGURE 12** Select PFAS concentrations with time for groundwater sampled within PAC PRZ. The hatched vertical line indicates time of injection. PAC, powdered activated carbon; PFAS, polyfluoroalkyl substances; PFBA, pentafluorobenzoic acid; PFOA, perfluorooctanoic acid; PFPeA, perfluoropentanoic acid; PRZ, permeable reactive zone [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



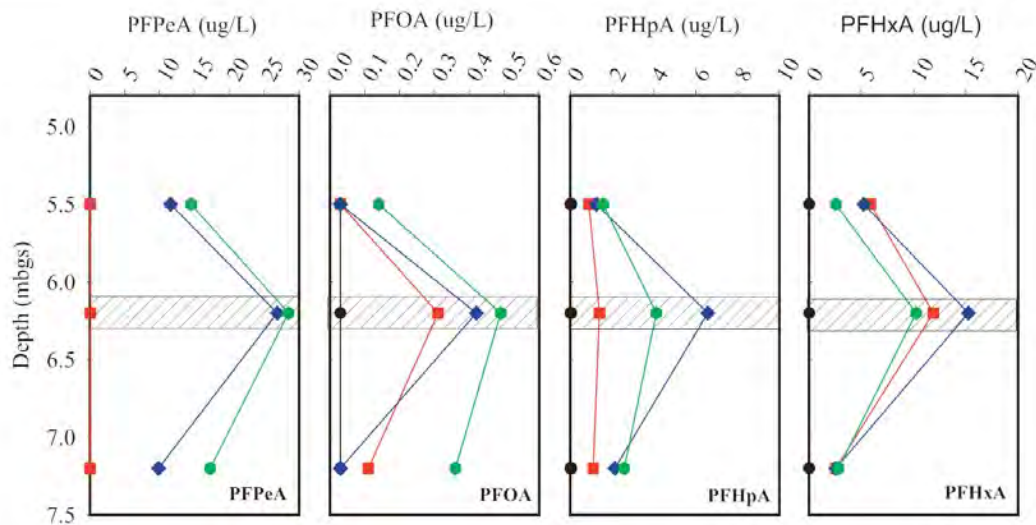
**FIGURE 13** Select PFAS removal percentages after 366 days of treatment within the six PRZs. CAC, colloidal activated carbon; IER, ion-exchange resin; PAC, powdered activated carbon; PFAS, polyfluoroalkyl substances; PRZs, permeable reactive zones

groundwater to below their respective detection limits (Figure 12). On Day 184, PFHxA and PFPeA were detected in groundwater at concentrations <25% of the influent concentration suggesting that these two PFAS were starting to break through the PAC (Figure 12). As with the IER and biochar, the shorter carbon chain PFAS broke through the PAC before the longer carbon chain PFAS, indicating possible preferential adsorption and/or competitive adsorption of the PFAS. After 366 days of treatment, concentrations of PFHpA and PFOA were detected within the groundwater along with PFHxA and PFPeA suggesting that the PAC was nearing its attenuation capacity (Figures 12 and 13). The results of the groundwater samples collected within the PAC PRZ on Day 549 indicated that the PFHxA and PFPeA had completely broken through the PAC PRZ, the PFHpA and PFOA were being partially attenuated, and PFNA and PFBA were still completely attenuated by the PAC (Figure 12). As with the IER and biochar PRZs, vertical sampling of the PFAS plume on Day 366 indicated higher concentrations of the PFAS within the coarse-grained sand seam compared with the surrounding finer-grained sand (Figure 14).

#### 4 | SUMMARY

Six pilot tests were conducted in an unconfined sandy aquifer to determine if a variety of oxidant and adsorptive reagents could effectively treat dissolved PFAS in situ over a 1.5-year period. The tests involved injecting six reagents; hydrogen peroxide, sodium persulfate, IER, biochar, PAC, and CAC using DPT and subsequently monitoring the groundwater for up to 549 days. The reagents were injected on a 1.5-m grid over multiple vertical intervals to form a PRZ ~3 m thick in the direction of groundwater flow.

The test results for the adsorptive-based PRZs (CAC, PAC, IER, and biochar) indicated that after 92 days following treatment the concentrations of the six PFAS detected within the influent groundwater were all below their respective detection limits, thereby, indicating that the reagents were effective at attenuating the PFAS over the short term. Monitoring of the groundwater over time indicated that the adsorptive capacity of the PAC, IER, and biochar reagents was partially exhausted by Day 184 for the biochar and PAC PRZs and by Day 278 for the IER PRZ with the shorter carbon chain PFAS showing partial or complete break through.



**FIGURE 14** Select PFAS dissolved concentrations with depth after 366 days. Red squares (■) represent PFAS within PAC PRZ, blue diamonds (◆) represent PFAS concentrations with the biochar PRZ, green hexagons (⬡) represent PFAS concentrations within the IER PRZ and black circles (●) represent PFAS concentrations within the CAC PRZ. The hatched area represents the coarse-grained sand seam. CAC, colloidal activated carbon; IER, ion-exchange resin; PAC, powdered activated carbon; PFAS, polyfluoroalkyl substances; PFHpA, perfluoroheptanoic acid; PFHxA, perfluorohexanoic acid; PFOA, perfluorooctanoic acid; PFPeA, perfluoropentanoic acid; PRZs, permeable reactive zones [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

These results suggest that competitive adsorption is occurring on the reagents with higher carbon chain PFAS being preferentially adsorbed compared to shorter carbon chain compounds such as PFPeA and PFHxA. Monitoring of the groundwater within the CAC PRZ failed to detect any of the PFAS for the monitoring period, including in groundwater sampled on Day 549, suggesting that the adsorptive capacity of the CAC had not been exhausted.

Measurements of the reagents within the aquifer postinjection suggest that CAC and persulfate were most uniformly distributed within the target injection zone with 100% of the aquifer solid and pore water samples containing CAC and persulfate, respectively. The concentration of TOC within the CAC-injected aquifer solids was six times greater than the TOC concentration in the aquifer solids outside of the targeted injection zone. Similarly, the persulfate concentration within the targeted injection zone was 4.5 orders of magnitude greater than the concentration of persulfate within the pore water outside of the targeted injection zone 24 hr after injection. Sampling of the aquifer cores for TOC within the PAC and biochar PRZs indicated that the respective reagents were preferentially distributed within the coarse-sand seam with concentrations of the reagents being measured within the targeted injection zone at a concentration of 518% and 344% greater within the sand seam than within the surrounding fine-sand aquifer for the PAC and biochar PRZs, respectively. These results suggest that consideration should be given to evaluating the mass flux of short-carbon chain PFAS as well as adsorptive competition when designing remedial programs based on mass flux.

Measurements of the TOC within the sand packs of monitoring wells installed before the injection of the reagents indicated

preferential accumulation of TOC within the sand packs of the monitoring wells located within the PAC and biochar PRZs. The mean TOC concentration within the PAC and biochar sand packs were 77% and 85% greater than the mean TOC concentration within the surrounding target injection zones. The mean TOC concentration within the sand pack of the monitoring well located in the CAC PRZ was determined to be 19% lower than the mean TOC concentration of the surrounding target injection zone suggesting that the CAC did not preferentially accumulate in the sand pack. Based on these measurements, consideration should be given to installing monitoring wells following the injection of coarse-grained reagents like PAC and biochar to minimize the possibility of bias results when groundwater sampling.

Posttreatment groundwater monitoring over 1.5 years within the six PRZs indicated that the PFAS was not effectively treated by either persulfate nor hydrogen peroxide and, in some cases, individual PFAS increased in concentration. Concentrations of PFAS in groundwater sampled within the PAC, CAC, IER, and biochar PRZs immediately after injection decreased to below their respective method detection limits; however, over time concentrations of individual PFAS within the groundwater sampled from the PAC, IER, and biochar PRZs exhibited partial or complete breakthrough. The only PRZ that completely attenuated the PFAS analyzed during the 1.5-year long study was the CAC PRZ. The increase in treatment performance for the CAC compared to the other adsorption based PRZs is attributed to better distribution of the CAC and increased capacity for the adsorption of the PFAS compared with the IER, PAC, and biochar used in this study.

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## Corrigendum

In Volume 30, in the article, "Six Pilot-Scale Studies Evaluating the *In Situ* Treatment of PFAS in Groundwater" by Rick McGregor the following changes were made

- 1) In the abstract the units for the concentration of various PFAS compounds were reported as ug/L, they should be ng/L and read:  
"24,000 ng/L perfluoropentanoic acid (PFPeA), up to 6,200 ng/L pentafluorobenzoic acid (PFBA), 16,100 ng/L perfluorohexanoic acid (PFHxA), up to 6,080 ng/L perfluoroheptanoic acid (PFHpA),

up to 450 ng/L perfluorooctanoic acid (PFOA) and up to 140 ng/L perfluorononanoic acid (PFNA)"

- 2) In the abstract pentafluorobenzoic acid should be read "perfluorobutanoic acid"
- 3) On page 41 pentafluorobenzoic acid should be changed to "perfluorobutanoic acid".

These errors were introduced during the writing process and do not affect any of the results presented.