RESEARCH ARTICLE

In Situ treatment of PFAS-impacted groundwater using colloidal activated Carbon

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Abstract

Poly- and perfluoroalkyl substances (PFASs) have been identified by many regulatory agencies as contaminants of concern within the environment. In recent years, regulatory authorities have established a number of health-based regulatory and evaluation criteria with groundwater PFAS concentrations typically being less than 50 nanograms per liter (ng/L). Subsurface studies suggest that PFAS compounds are recalcitrant and widespread in the environment. Traditionally, impacted groundwater is extracted and treated on the surface using media such as activated carbon and exchange resins. These treatment technologies are generally expensive, inefficient, and can take decades to reach treatment objectives. The application of *in situ* remedial technologies is common for a wide variety of contaminants of concern such as petroleum hydrocarbons and volatile organic compounds; however, for PFASs, the technology is currently emerging. This study involved the application of colloidal activated carbon at a site in Canada where the PFASs perfluorooctanoate (PFOA) and perfluorooctane sulfonic acid (PFOS) were detected in groundwater at concentrations up to 3,260 ng/L and 1,450 ng/L, respectively. The shallow silty-sand aquifer was anaerobic with an average linear groundwater velocity of approximately 2.6 meters per day. The colloidal activated carbon was applied using direct-push technology and PFOA and PFOS concentrations below 30 ng/L were subsequently measured in groundwater samples over an 18-month period. With the exception of perfluoroundecanoic acid, which was detected at 20 ng/L and perfluorooctanesulfonate which was detected at 40 ng/L after 18 months, all PFASs were below their respective method detection limits in all postinjection samples. Colloidal activated carbon was successfully distributed within the target zone of the impacted aquifer with the activated carbon being measured in cores up to 5 meters from the injection point. This case study suggests that colloidal activated carbon can be successfully applied to address low to moderate concentrations of PFASs within similar shallow anaerobic aquifers.

1 | INTRODUCTION

Poly- and perfluoroalkyl substances (PFASs) have been identified as emerging contaminants and have attracted concern from regulatory bodies over the past 20 years because they are widespread and persistent in the environment, have potential for bioaccumulation, and may have adverse effects on the immune system, liver, and development of children/fetuses (U.S. Environmental Protection Agency [EPA], 2009; Environment and Climate Change Canada [ECCC], 2017a). These compounds are used in metal plating, firefighting, photography, and aviation industries for applications including fume suppressants, foaming agents, and hydraulic fluid additives (Hunter-Anderson, Long, Porter, & Anderson., 2016; Government of Canada, 2008). PFASs are no longer produced in Canada (ECCC, 2016) or the United States

(ECCC, 2017b), but can be imported from China as of 2003 (Butt, Berger, Bossi, & Tomy, 2010). Canada has no current drinking water or groundwater regulations for any PFAS; however, the Federal Soil Quality Guidelines and Groundwater Quality Guidelines for PFOS indicate 0.21 milligrams per kilogram (mg/kg) for fine soil, 0.14 mg/kg for coarse soil, and 68 micrograms per liter (μg/L) for groundwater for the protection of freshwater life (ECCC, 2017b). The EPA drinking water health advisory level for the sum of perfluorooctanoate (PFOA) and perfluorooctanesulfonate (PFOS) concentrations is 70 nanograms per liter (ng/L), while other jurisdictions pose stricter regulations (NGWA, 2017).

The remediation of PFASs is challenging for many reasons, including the highly recalcitrant nature of these compounds which is likely due to multiple stable fluoride-carbon bonds (National Ground Water
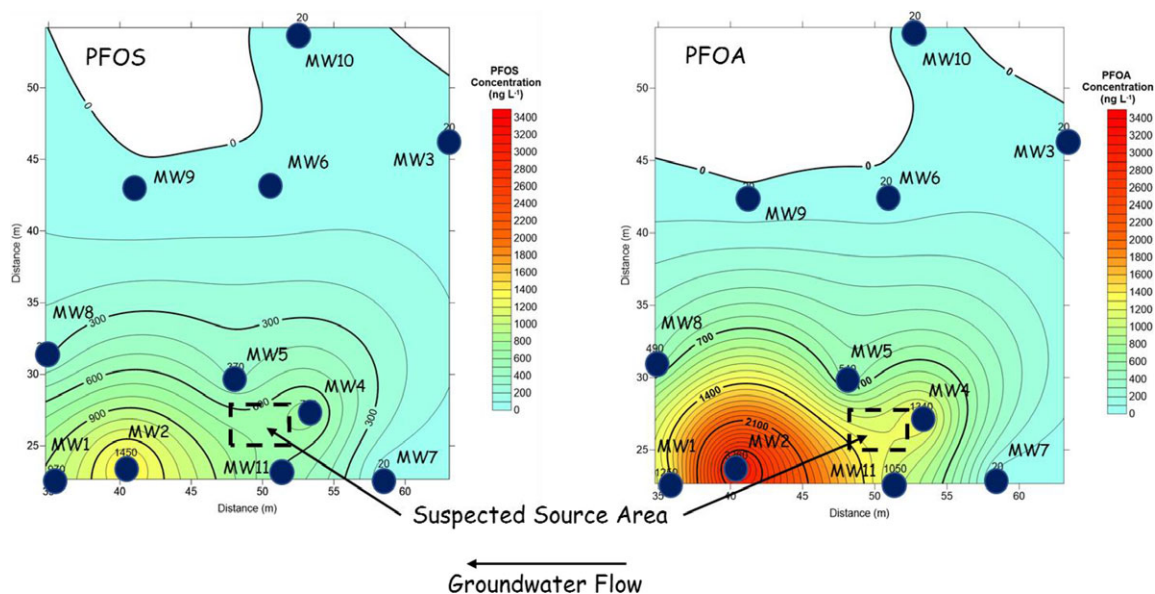
EXHIBIT 1 Site map showing monitoring well locations and dissolved concentrations of PFOA and PFOS

Association [NGWA], 2017), low or uncertain regulatory guidelines, complicated environmental fate (Ferrey et al., 2012), and limited proven remediation techniques. Groundwater PFOS concentrations at commercial and industrial sites used for firefighting training purposes range from below detection limit to 560 μg/L (ECCC, 2017b). To conform to the strictest US regulations, the most heavily contaminated Canadian sites would have to achieve >99.6 percent removal to meet the applicable target concentrations. Some precursors are less mobile and undergo transformation reactions to produce other PFASs by aerobic biodegradation (McGuire et al., 2014), which could act as a source of dissolved phase contamination for up to 18 years (Weber, Barber, Leblanc, Sunderland, & Vecitis, 2017).

PFOS has been successfully remediated with *ex situ* techniques such as soil washing, activated carbon filtration, ion exchange, incineration, sonic destruction, advanced oxidation, or advanced reduction activated by ultraviolet radiation (Hale et al., 2017; Merino et al., 2016). Many of these techniques are cost-prohibitive, energy-intensive, produce toxic by-products, and may be unsuitable for *in situ* application. Activated carbon is a promising sorbent amendment with documented treatment efficiencies of 93.9 to 99.9 percent (Hale et al., 2017). The *in situ* application of activated carbon has the potential to meet current and future regulatory objectives as powdered activated carbon and colloidal activated carbon have smaller particle sizes and more available surface area than granulated activated carbon used in filtration. Increased surface area provides increased sorption sites and PFAS sorption improves as particle size decreases, because smaller particles have a greater ratio of surface area to mass (Bao et al., 2014; Ochoa-Herrera & Sierra-Alvarez, 2008; Pramanik, Pramanik, & Suja, 2015; Qu et al., 2009; Yu et al., 2009). The application of colloidal activated carbon is a sorption technique, which removes PFASs from the aqueous phase and into the solid phase.

This case study describes the *in situ* treatment of PFAS compounds with colloidal activated carbon in a shallow, thin, silty sand aquifer with short and moderate term monitoring of PFAS, microorganisms, and geochemical parameters.

2 | STUDY SITE

The field study was completed at a site in Central Canada where fire-training exercises were conducted during the 1970s and 1980s. Reportedly, PFAS-containing firefighting foam was used during the exercises, which resulted in PFAS contamination in the shallow groundwater. The shallow geology of the site is composed of medium sand containing some silt with occasional discontinuous sand lenses that are approximately 0.02 meters (m) thick.

Groundwater at the site is encountered at a depth of approximately 1 m below ground surface. Based on a horizontal hydraulic gradient of 0.06 m/m and a mean horizontal hydraulic conductivity (K_H) of 2.6 m a day a Darcy Flux of 0.8 m a day is estimated for the unconfined aquifer.

The PFOS plume was estimated to cover an area of 780 m^2, whereas the PFOA plume was estimated to cover an area of 700 m^2, both extending from the surface of the water table ($\sim$0.9 m below ground surface) to approximately 1.7 m below ground surface (Exhibit 1). A total mass of 0.94 mg of PFOS and 0.84 mg of PFOA was estimated to be present within the groundwater in the unconfined aquifer.

3 | METHODOLOGY

The performance-monitoring program involved a combination of soil and groundwater sampling. A total of nine boreholes were advanced using dual-tube sampling methods to collect continuous cores of the soil to determine the distribution of the colloidal activated carbon and soil samples to estimate the K_H of the aquifer. A total of seven 0.05 m diameter PVC wells equipped with a 3.0 m-long slot 10 screen,
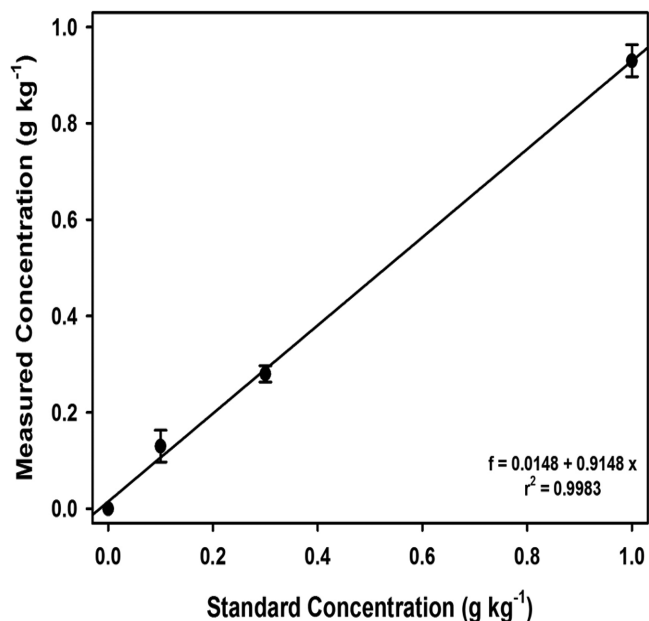
EXHIBIT 2 Calibration curve for known standards of colloidal activated carbon and measured TOC

screened across the water table, were installed for the collection of groundwater samples and monitoring of hydrogeological parameters prior to and following injection of the colloidal activated carbon.

Continuous cores of the aquifer materials were collected at 13 locations and sampled at 0.15 m vertical intervals for grain size analysis. The grain size distribution was used to estimate the K_H of the aquifer using the method outlined by Kozeny (1927) and Carmen (1956). Rising-head tests were completed on three monitoring wells to estimate the horizontal hydraulic conductivity (K_H) of the aquifer and to determine the variability in K_H across the study area. The tests were completed by quickly lowering the water level in the subject wells and then monitoring the water level recovery over time. Estimates of K_H were calculated using the method of Bouwer and Rice (1976) for unconfined aquifers.

Aquifer samples were analyzed for the fraction of organic carbon (f_{oc}) and total organic carbon (TOC) using the methods of Walkley (1947) and ASTM (2011) by combustion using a LECO furnace (ASTM E1019 and ASTM E1409, 2013). Cores of aquifer material were collected and analyzed for f_{oc} and TOC prior to the injection of the colloidal activated carbon to determine if the analyses were able to separate the two forms of carbon (i.e., f_{oc} versus TOC). A four-point standard curve was created to validate the LECO combustion method by analyzing three spiked samples of colloidal activated carbon mixed with Ottawa quartz sand and analyzing a blank sample of Ottawa quartz sand. The samples were analyzed in triplicate. The data fit a linear regression ($R^2 = 0.9983$, Exhibit 2).

Groundwater samples were collected during the field study over an 18-month period to provide baseline and treatment performance data over time. The groundwater was collected using low-flow sampling methods (Puls and Barcelona, 1996). Oxidation–reduction potential (ORP), pH, temperature, specific conductivity, turbidity, and dissolved oxygen (DO) were measured at the wellhead using a flow-through cell connected to a YSI Professional Plus handheld multiparameter meter. The pH meter was calibrated with buffers of 4.0, 7.0, and 10.0 standard units prior to each sampling event and verified between each sample. The ORP was verified with Zobell's solution (Nordstrom, 1977) prior to each well measurement, whereas specific conductivity was calibrated to a National Institute of Standards and Technology (NIST) traceable standard solution.

Samples for PFOA and PFOS analyses were collected on five occasions whereas samples for PFOA, PFOS, perfluorobutanesulfonate (PFBS), perfluorohexanesulfonate (PFHxS), perfluorodecanesulfonate (PFDS), perfluorooctanesulfonamide (PFOSA), perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorononanoic acid (PFNA), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnA), and perfluorododecanoic acid (PFDoA) were analyzed during the 18-month sampling event. The samples were collected in high-density polyethylene bottles using dedicated polyethylene tubing. Samples were analyzed using liquid chromatography-mass spectrometry (LC/MS, Paracel Laboratories Ltd., Ottawa, Ontario).

Samples for the analyses of inorganic parameters chloride (Cl), sulfate (SO_4), nitrate (NO_3), dissolved and total iron (Fe), dissolved and total manganese (Mn), and alkalinity were collected. The total Fe, total Mn, SO_4, NO_3, and Cl samples were not filtered, whereas alkalinity, dissolved Fe, and dissolved Mn were filtered with a 0.45-micron cellulose acetate membrane. Total and dissolved Fe and Mn were acidified to a pH below 2 with nitric acid (HNO_3, Paracel Laboratories Ltd., Ottawa, Ontario). In addition to chemical parameters, microorganism analyses were completed on samples collected during the 18-month sampling event. The analyses included DNA amplification using quantitative polymerase chain reaction (qPCR) and sequencing and clustering analyses. The sequencing was completed using a MiSeq system by loading 7 pico-mole aliquot of sample spiked to 5 percent PhiX for sequencing control. The clustering was completed using UPARSE (Gene-Trac® NGS, SiREM, Guelph, Ontario).

The injection event was conducted over a two-day period using a combination of colloidal activated carbon and oxygen-releasing material (Regenesis, San Clemente, CA). The oxygen-releasing material was added to address the dissolved petroleum hydrocarbon compounds also present within the groundwater. A total of 290 kg of colloidal activated carbon and 176 kg of oxygen-releasing material was mixed with 3,120 L of water and injected into 20 temporary direct-push locations at pressures of less than 25 psi.

4 | RESULTS AND DISCUSSION

4.1 | Horizontal hydraulic conductivity

The K_H was estimated with depth and it varied from 0.6 to 8.8 m a day with the highest K_H associated with the sand lens located at approximately 1.5 m below ground surface (Exhibit 3). The K_H within the target zone, 0.9 m to 1.7 m below ground surface, varied from 1.6 m to 8.8 m a day whereas the K_H outside of the target zone varied from 0.6 m to 2.9 m a day. These values are comparable to the values calculated from

Kh (m/day)
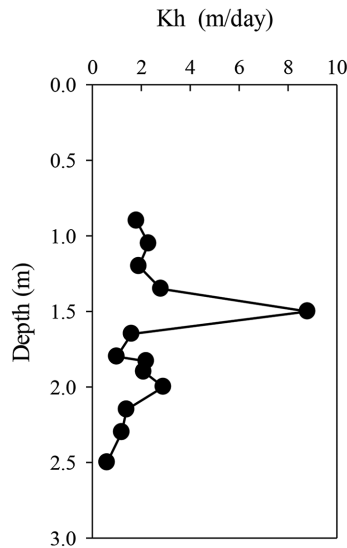
EXHIBIT 3 Horizontal hydraulic conductivity (Kh) versus depth

Organic C (g/kg)   foc (g/kg)
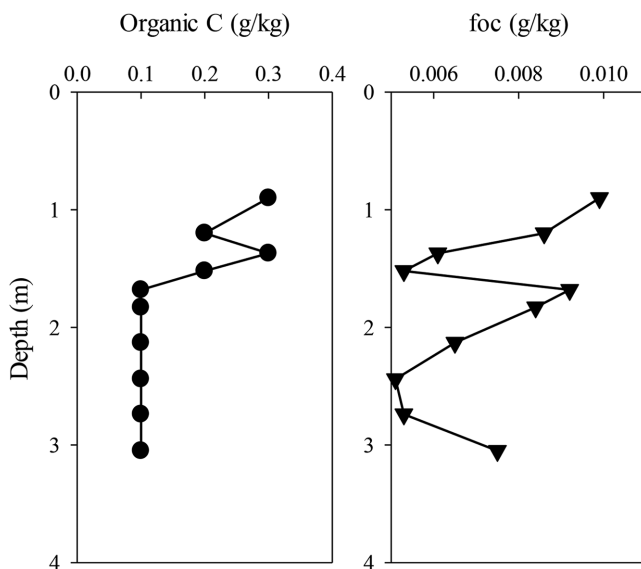
EXHIBIT 4 Vertical profiles for total organic carbon (TOC) and fraction organic carbon (f_{oc}) within aquifer material prior to the injection of colloidal activated carbon

rising-head tests completed on three of the monitoring wells, which ranged from 2.9 m to 9.2 m a day.

4.2 | Distribution of activated carbon

Prior to injection, all aquifer core samples within the target injection zone (i.e., 0.9 m to 1.7 m below ground surface) analyzed for TOC were below the method detection limit of 0.1 gram per kilogram (g/kg) (Exhibit 4). Postinjection aquifer cores were collected to evaluate the vertical and lateral distribution of the activated carbon. The cores were collected 24 hr after injection and analyzed for TOC. To evaluate the radius of influence of colloidal activated carbon, cores were collected 0.75 m, 1.52 m, 3.05 m, and 4.57 m away from an injection point (Exhibit 5). Soil samples within the cores were collected every 0.15 m to 0.30 m vertically from 0.91 m to 3.00 m below ground surface to eval-

uate the vertical distribution of the TOC. The ideal vertical distribution of TOC as activated carbon is within the zone of impacts, which was determined to be between 0.90 m and 1.70 m below ground surface. Total organic carbon was detected at least 4.57 m away from the injection point with a maximum concentration of 0.2 g/kg measured in this core at depths of 1.20 m and 1.35 m below ground surface. The highest TOC concentration, 0.4 g/kg, was detected 1.52 m away from the injection point at a depth of 1.37 m (Exhibit 6).

Five cores, distributed across the study site, were collected at midpoints between injection points to evaluate the distribution of the activated carbon over the site-wide injection area both vertically and laterally (Exhibit 5). Total organic carbon, assumed to be activated carbon, was found in all five core samples with a maximum concentration of 0.4 g/kg detected in Core 4 at a depth of 1.52 m below ground surface (Exhibit 7). Comparison of the percentage of aquifer samples that had TOC detected versus the percentage of samples in which the TOC was below the detection limit (0.1 g/kg) indicated that 92 percent of the samples within the target injection zone (i.e., 0.90 m to 1.70 m below ground surface) had measurable TOC, assumed to be activated carbon, detected at concentrations greater than the method detection limit of 0.1 g/kg. Only 2 of the 25 samples did not have detectable TOC present.

Overall, detectable TOC was measured in 84.4 percent of the samples collected ($n = 45$) within the target injection zone whereas only 4.4 percent of the samples outside of the target zone had total organic carbon detected ($n = 45$). Following the injection, samples collected within the target injection zone had a mean TOC of 0.23 g/kg and aquifer samples collected outside of the target injection zone had a mean TOC concentration of 0.104 g/kg. The mean f_{oc} of the samples within the target injection zone was 0.0078 versus a mean of 0.00066 outside of the target injection zone.

These results indicate that the colloidal activated carbon was successfully injected within the target injection zone with a site-specific radius of influence of greater than 4.57 m.

5 | TREATMENT OF PFOA AND PFOS

Groundwater samples for PFOA and PFOS were collected prior to the injection of the colloidal activated carbon and 3, 6, 9, 12, and 18 months postinjection. Initially, PFOA and PFOS were detected within 11 monitoring wells and 6 were selected for long-term monitoring (Exhibit 8). Wells in which PFOS was detected had concentrations ranging from 280 ng/L (MW8) to 1,450 ng/L (MW2) whereas wells in which PFOA was detected had concentrations from 490 ng/L (MW8) to 3,260 ng/L (MW2) prior to the injection of the colloidal activated carbon (Exhibit 8). Based on these concentrations, it is likely that the source of PFAS was located upgradient of MW2.

Postinjection groundwater concentrations of PFOA and PFOS from samples collected from six monitoring wells were all below the method detection limits (MDLs) of 20 and 30 ng/L for PFOA and PFOS, with the exception of MW1 with PFOS detected at 40 ng/L (MDL = 30 ng/L) in the groundwater sampled 18 months after the colloidal activated carbon injection (Exhibit 8). These results suggest that PFOA and PFOS
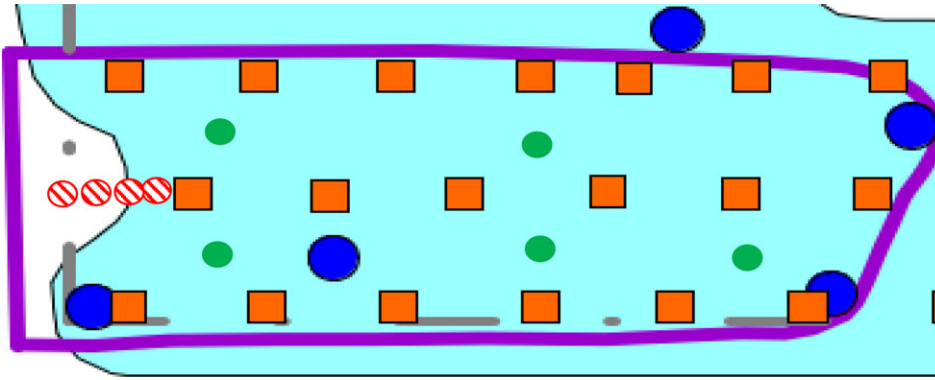
EXHIBIT 5 Borehole locations to evaluate radius of influence (⊘) and overall coverage (●) as well as showing direct push injection grid (▧)
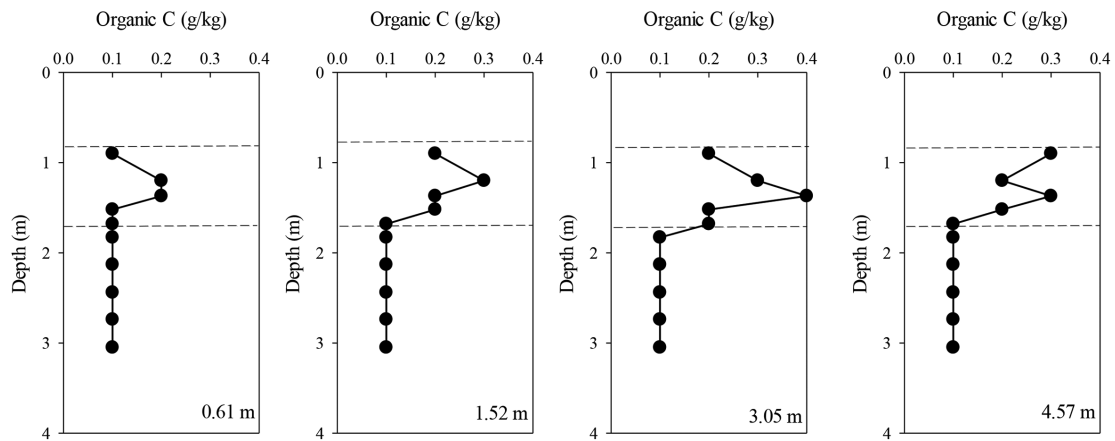
EXHIBIT 6 Vertical profiles for TOC showing radius of influence from direct push injection location located 0.61 m, 1.52 m, 3.05 m, and 4.57 m away from profiles. Dashed lines represent zone of PFAS groundwater impacts
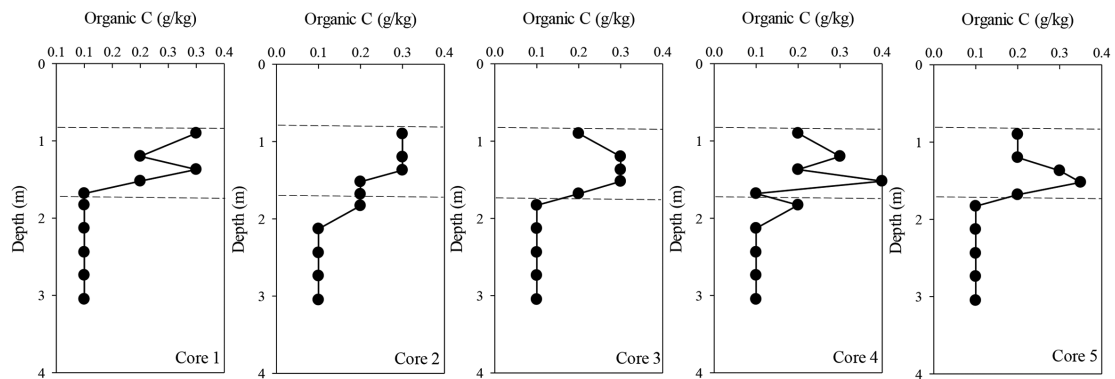
EXHIBIT 7 Vertical profiles for TOC showing overall coverage of colloidal activated carbon within injection area. Dashed lines represent zone of PFAS groundwater impacts

are effectively attenuated by the colloidal activated carbon over the short and moderate term.

5.1 | Treatment of other PFAS

Groundwater samples for 12 other PFAS were collected from monitoring wells within the PFAS plume during the 18-month postinjection sampling event. PFASs analyzed for included PFBS, PFHxS, PFDS, PFOSA, PFBA, PFPeA, PFHxA, PFHpA, PFNA, PFDA, PFUnA, and PFDoA. These compounds represent a mixture of shorter carbon chain

and longer carbon chain PFASs. It would be expected that the shorter carbon chain PFASs, such as PFBS and PFBA, would breakthrough prior to the longer chain PFASs, such as PFUnA and PFDoA. After 18 months of *in situ* treatment, all the PFASs listed previously were below their respective MDLs, with the exception of PFUnA, which was detected at 20 ng/L (MDL = 20 ng/L) in the groundwater samples collected from MW1. As a longer chain PFAS, preferential adsorption of PFUnA ($C_{11}HF_{21}O_2$) over shorter carbon chain PFASs onto the activated carbon is likely and further sampling is required to confirm the presence of PFUnA in MW1.
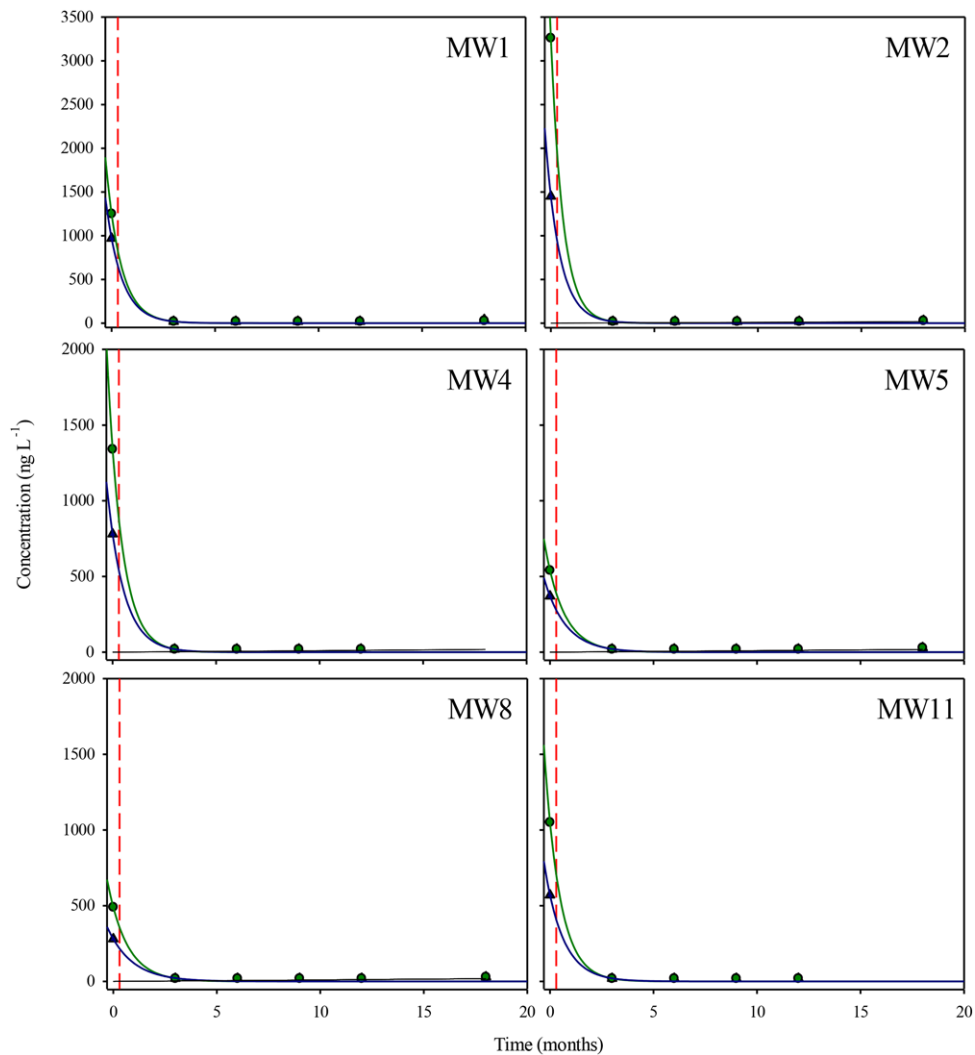
EXHIBIT 8 PFOS (▲) and PFOA (●) groundwater concentrations with time. Dashed vertical line represents the injection of colloidal activated carbon

5.2 | Microorganism analyses

Groundwater samples from three monitoring wells (MW2, MW5, and MW9) were collected during the 18-month postinjection sampling event and analyzed for a variety of parameters including quantification of total biomass by qPCR, 16S rRNA gene sequencing and cluster analyses. Two of the monitoring wells sampled are located within the PFAS plume (MW2 and MW5) whereas MW9 is located outside of the PFAS plume but within the petroleum hydrocarbon plume.

The 16S rRNA gene copies within each of the three samples were similar with between 1×10^7 and 3×10^7 copies per liter being determined for *Archaea* and between 1×10^9 and 4×10^9 copies per liter being determined for *Bacteria* within the groundwater (Exhibit 9). The microbial diversity of the microorganisms was inferred by analyzing the operational taxonomic units (OTUs) within each groundwater sample, which are proportional to microbial diversity. The groundwater collected from MW2, which is located within the core of the PFAS plume, had the greatest number of OTUs at approximately 2,636 whereas groundwater collected from MW5 and MW9 had approximately 1,790 OTUs and 1,572 OTUs, respectively (Exhibit 10). These

EXHIBIT 9 Summary of DNA extraction results along with the total *Bacteria* and *Archaea*

Well Number	Total DNA Extracted (ng)	*Archaea* 16S rRNA (gene copies/L)	*Bacteria* 16S rRNA (gene copies/L)
MW2	378	2×10^7	1×10^9
MW5	1,041	1×10^7	2×10^9
MW9	467	3×10^7	4×10^9

values suggest that the microbial diversity within the PFAS plume exceeds the diversity outside of the PFAS plume.
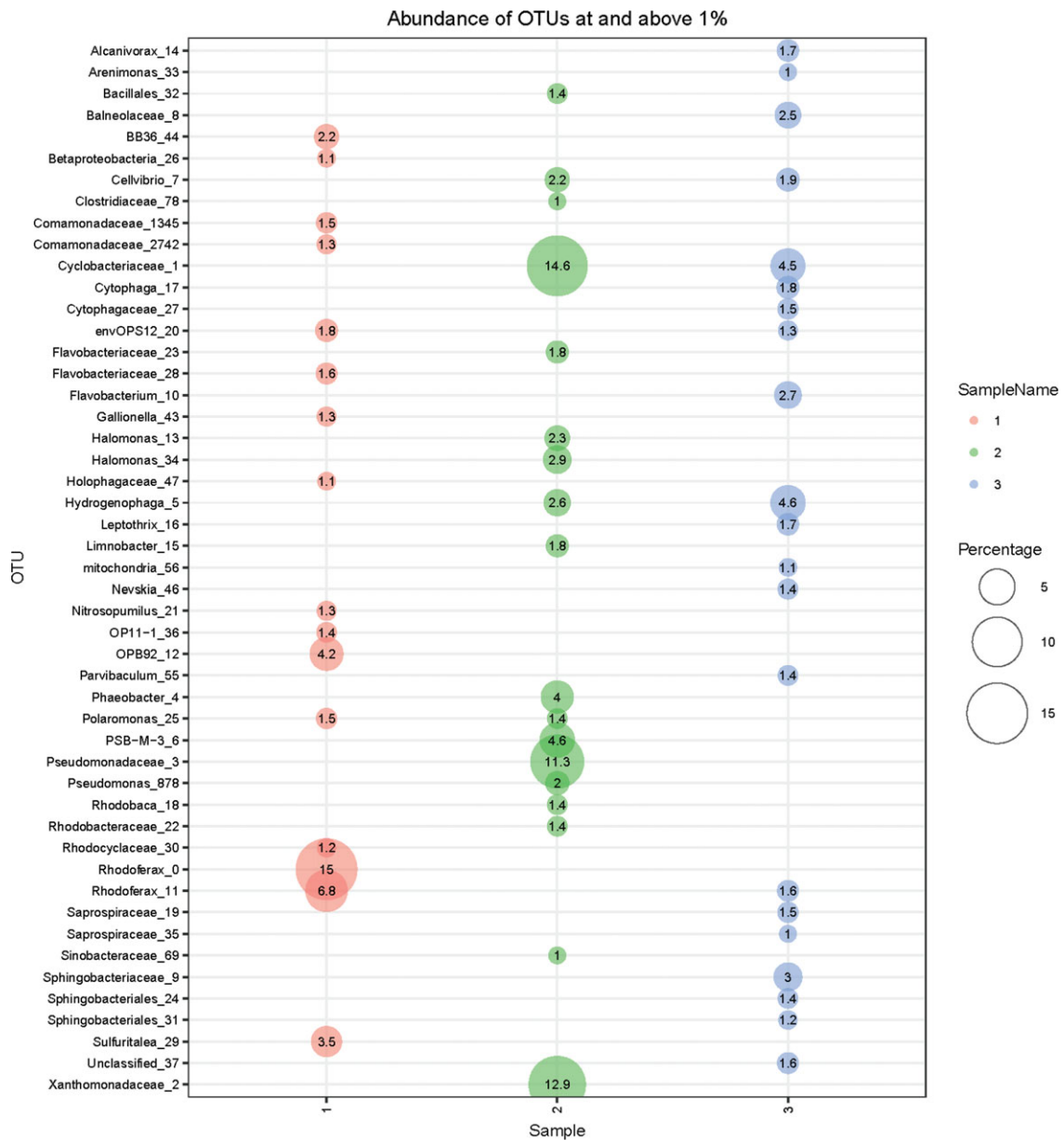
An evaluation of the taxonomic affiliation and relative abundance of the microbial community indicates that many of the taxa are affiliated with aerobic or mildly reducing conditions with a general absence of significant proportions of sulfate reducers or methanogens which indicate more reducing conditions. Within the core of the PFAS plume (i.e., monitoring wells MW2 and MW5), *Rhodoferax*, *OPB92*, and *Sulfuritalea* are predominant; whereas, on the fringe of the PFAS plume, *Hydrogenophaga*, *Cyclobacteriaceae* were somewhat
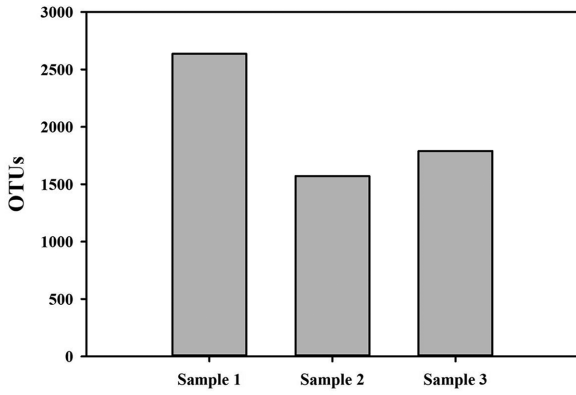
EXHIBIT 10 Number of operational taxonomic units (OTUs) identified in each sample, which is a representation of the diversity of microorganisms present. Samples 1, 2, and 3 correspond with MW2, 9, and 5, respectively

proportionally higher within an even distribution of various taxa. Outside of the PFAS plume and within the petroleum hydrocarbon plume, *Cyclobacteriaceae* and *Pseudomonadaceae* and *Xanthomonadaceaea* were dominant (Exhibit 11).

Rhodoferax is a gram-negative genus often associated with aerobic environments such as those that may exist within petroleum hydrocarbon plumes injected with oxygen as was performed at this site. *Pseudomonadaceae/Pseudomonas* are gram-negative and have been associated with the degradation of petroleum hydrocarbons (Jain et al., 2011) and been implicated in defluorination reactions, although not of perflourinated compounds (Key, Howell, & Criddle, 1998). Also, of note is the presence of *Polaromonas* which is a genus that is often comprised of psychrophilic (cold loving) bacteria associated with the aerobic degradation of various compounds including cis-1,2-dichloroethene and naphthalene (Sierra-Garcia et al., 2014).

EXHIBIT 11 Bubble plot demonstrating taxonomic affiliation and relative abundance of key taxa. Number and relative size of bubble denotes the percentage of taxa as proportion of total microbial community. Samples 1, 2, and 3 correspond with MW2, 9, and 5, respectively

6 | CONCLUSIONS

The remediation of PFASs in groundwater is a challenge due to a variety of factors including the limited understanding of the fate and transport of PFASs, the number of PFASs, the compounds' resistance to biological and chemical degradation, and the low remedial concentrations required to meet environmental standards (i.e., in general, less than 100 ng/L). Historically, PFAS-impacted groundwater has been remediated using pump and treat methods utilizing a variety of media for removal, including activated carbon, ion-exchange resin, and membranes. These methods, while effective, tend to be expensive and must be operated and maintained for a period of years to decades due to limitations associated with the geology and hydrogeology of the impacted aquifers. Applications of *in situ* treatment of PFAS in groundwater have been limited to laboratory and small-scale field studies. Currently, the results of these studies have shown limited success.

Colloidal activated carbon is one potential *in situ* technology for the treatment of PFAS-impacted groundwater. This field study utilized colloidal activated carbon for the treatment of petroleum hydrocarbons within a shallow unconfined aquifer. Within the petroleum hydrocarbon plume, the perflourinated compounds PFOA and PFOS were detected at concentrations greater than the recommended advisory limits. The injection of colloidal activated carbon resulted in the concentration of PFOA decreasing from a maximum of 3,260 ng/L to less than the MDL over an 18-month period. The PFOS concentration decreased from a maximum of 1,450 ng/L to less than the MDL limit in all of the groundwater samples collected from monitoring wells within the PFAS plume with the exception of one well sampled 18 months postinjection in which the PFOS was detected at 40 ng/L (MDL = 30 ng/L). Analysis of other PFASs in groundwater samples collected from the five-well monitoring network during the 18-month postinjection sampling event did not detect PFBS, PFHxS, PFDS, PFOSA, PFBA, PFPeA, PFHxA, PFHpA, PFNA, PFDA, PFUnA, and PFDoA at concentrations greater than their respective MDL, with the exception of PFUnA which was detected at its detection limit in a sample from one monitoring well.

The distribution of the colloidal activated carbon was assessed using cores from the aquifer. Activated carbon was effectively distributed with radii of influence of greater than 4.57 m. Furthermore, the colloidal activated carbon at this site was effectively injected using direct push technology into the target impacted zone within the aquifer with 92 percent of the samples collected within the target zone having detectable concentrations of TOC.

Microbial analyses of the groundwater 18 months postinjection indicate that the microbial community within the PFAS plume is more diverse than the community outside of the plume which suggests that the PFAS may not be toxic to microbes and that there is potential for biodegradation.

The results of this field application suggest that colloidal activated carbon may be an effective treatment medium for low to moderate dissolved concentrations of PFOS, PFOA, and other PFASs *in situ*. The effectiveness of *in situ* PFAS treatment by colloidal activated carbon requires further monitoring to determine if the colloidal activated carbon can be effective over the long term.

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