EVALUATION OF THE TREATMENT OF GROUNDWATER ARSENIC AT MINING AND INDUSTRIAL SITES USING ZVI AND BOFS PERMEABLE REACTIVE BARRIERS

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Abstract: Permeable reactive barriers installed at a gold mine and at a chemical manufacturing facility have been effectively remediating arsenic contaminated groundwater since they were installed in 2002. At the mine site, the PRB is pilot scale and uses a reactive mixture consisting of zero valent iron filings and organic carbon derived from locally available wood chips. For experimental control the PRB is isolated from the surroundings by sheet pilings and As-contaminated groundwater from tailings at the mine is pumped through the reactive materials at controlled rates. For more than four years, As input at around 20 mg/L has been treated to <0.01 mg/L. At the chemical manufacturing site the PRB is full-scale and the reactive material is entirely granular basic oxygen furnace slag. This full-scale PRB consists of two parallel 600 m long trenches \textasciitilde5 m apart and installed across the direction of groundwater flow to a depth of approximately 10.5 m, where it intercepts up a meter of clay aquitard. Each PRB is approximately 0.6 m wide. For more than four years, As flowing into the PRB at 1-3 mg/L has been treated to <0.01 mg/L. The two PRB locations have very different groundwater chemistry and use dissimilar reactive materials, but both are providing high levels of As treatment. At the pilot scale PRB there is no indication that the level of As treatment is diminishing after four years of operation. The full-scale PRB locally shows signs of partial loss of reactivity in the upgradient PRB however, the downgradient PRB remains effective and a zone of low-As water now extends more than 6 m past the PRBs. The reactive materials from these PRBs have been tested to provide insight into the As removal mechanisms and factors that affect their performance.

Key Words: zero valent iron, basic oxygen furnace slag

Introduction

Arsenic is a contaminant in groundwater plumes at many mine sites, industrial sites and coal burning power generation stations. Arsenic (As) is toxic at high concentrations. At low concentrations As is a human carcinogen and suspected mutagen. Arsenic is common in many natural volcanic soils and in the byproducts of precious metal mining. Releases of As to the environment occur due to weathering of soils and mine tailings, and by the commercial use of As, including steel manufacture, the burning of coal, wood preservation and agricultural practices. The Canadian Interim Maximum Acceptable Concentration and the U.S. EPA acceptable limit for As in drinking water have recently been lowered to 10 ppb. In light of this new standard, there is increased incentive to find new alternatives to conventional groundwater treatment methods. Although current remediation technology is capable of meeting the new drinking water limit, the estimated cost to society for this treatment is significant. The reduction of As released from industrial properties, through the use of PRBs may result in lower economic costs to meet these

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Treatment goals.

Permeable reactive barriers (PRBs) are gradually being accepted as a remedial alternative for mining and industrial properties that have groundwater contamination problems. A PRB consists of a trench filled with reactive materials placed in the pathway of contaminated groundwater. The reactive materials are selected based on their contaminant-removal and permeability properties, as well as cost and availability. As contaminants flow through the reactive materials, they are degraded, precipitated or adsorbed to the reactive material, thus removing the contaminant from solution. Ideally, the reactive material will have a long lifetime of reactivity and will remove and retain the contaminants permanently. Treatment in PRBs is passive, in that mechanical pumping, chemical additions and manual labour beyond the installation of the PRB, are not required.

Over the past ten years, researchers conducting batch studies and laboratory and field column studies have demonstrated the use of several different reactive materials and removal mechanisms for the treatment of As-bearing groundwater. Zero valent iron (ZVI) filings have been evaluated in batch studies and in laboratory and field columns (Blowes et al., 1997; Gu et al., 1999; McRae et al., 1999; Farrell et al., 2001; Su and Puls, 2001; Nikolaidis et al., 2003) for removing Fe, U, Cr, As, Se and other metals from contaminated groundwater. In these studies, As was removed from solution by several different mechanisms, including coprecipitation with iron oxyhydroxides and green rust and adsorption to iron oxide surfaces. In a column study, Bain et al. (2003) evaluated the performance of a mixture of zero valent iron plus organic carbon, for the removal of As from sulfate-bearing mine-water. In that study, the proposed mechanisms of As removal are the same, with additional removal by As-sulfide precipitation, following sulfate reduction. Laboratory investigations conducted by the University of Waterloo (UW) and by DuPont indicate that basic oxygen furnace slag (BOFS) is also effective for the treatment of As-contaminated water.

BOFS is currently an inexpensive, readily available byproduct of steel manufacturing. It is composed of calcium silicates and ferrites (e.g. $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$) combined with fused oxides of iron, aluminum, manganese, calcium (e.g. portlandite) and magnesium. BOFS is granular and poorly sorted. Several characteristics of the BOFS that are favourable for the removal of As from water include its high surface area, high content of Al, Fe, Mg and Mn oxides and alkaline nature. While there are multiple other uses for BOFS in industry, the use of BOFS for groundwater remediation offers the steel making industry a new outlet for this material. The mechanisms of As removal from groundwater by BOFS are not well defined at this time. Due to the high surface area of iron oxides and the abundance of calcium in the BOFS, removal is presumed to be a combination of adsorption removal of arsenate and potentially the precipitation of calcium arsenate minerals.

Under field site conditions, variables such as the nature of the groundwater flow system in and around the PRB, as well as ambient geochemical conditions (pH, redox, temperature, solute load) affect the performance of the PRBs and the reactive materials. In this paper, we describe our investigation of four PRBs, installed at industrial sites in 2002 for treatment of As in groundwater and monitored for performance one to two times per year. Two of these PRBs contain mixtures of zero valent iron and organic carbon, and the other two contains BOFS. To our knowledge these are the first field-site installations of PRBs using these materials for the treatment of groundwater As.

Methods

To facilitate monitoring of the geochemistry within and up/downgradient, piezometers were installed at close spacing vertically and in the direction of flow (see Figures) in each of the PRBs. Detailed groundwater chemistry, including field measurements of pH, Eh, alkalinity, As speciation and sample collection for analysis of dissolved (filtered to 0.45 µm) cations (by ICP-MS and OES) and anions (by ion
chromatography) was done annually. Where possible, cores of the reactive material were collected near the piezometers for mineralogical analysis. The mechanisms responsible for As removal from the water are under investigation by means of sequential chemical extractions, geochemical modelling with MINTEQA2 (Allison et al., 1990) and mineralogical examination by SEM, x-ray fluorescence spectroscopy and synchrotron x-ray absorption spectroscopy. This information will support our evaluation of the long-term stability of the removed As and about the longevity of the reactive materials.

Results and Discussion

Full-Scale Basic Oxygen Furnace Slag PRB

In-house studies conducted by E.I. DuPont de Nemours and Company confirmed that As in groundwater at one of their industrial sites could be treated with BOFS. On the basis of these results, in June 2002 DuPont installed a full-scale (600 m long x 11.3 m deep) PRB composed of BOFS (100%, granular, unsorted) for the treatment of As-contaminated groundwater at an industrial site in East Chicago, Indiana. The installation consists of two parallel 76 cm wide PRBs, separated ~4.5 m in the direction of flow. Parallel PRBs were installed to minimize the potential of As transporting untreated past the PRBs. The PRBs were installed in a vacant part of the property using a trenching machine, which operated from a bench excavated ~3 m below grade. The PRB is installed from the base of a fill layer at the site (1.5 m) to a depth of 11.3 m, including 0.7 m penetration into a clay aquitard (Figure 1). The aquifer consists of aeolian fine sand. Rising head response tests indicate that the hydraulic conductivity of the PRBs is generally slightly greater than that of the aquifer ($10^{-5}$ vs $10^{-6}$ m/s). Hydrogeological conditions suggest that groundwater at the site flows at approximately 5-10 m/yr. At this velocity, groundwater should have traveled 15-30 m beyond the PRBs by 2005, although no geochemical indicators of groundwater velocity have been identified. The average residence time for water in each of the PRBs is about five weeks and between 20 and 40 pore volumes of water should have flowed through the PRB as of July 2005, when the last detailed dataset was collected.

Groundwater at the site is contaminated with 1-3 mg/L of As, predominantly as arsenite (As(III)). The groundwater is neutral-pH and slightly aerobic (Eh ~50 mV). Other dissolved constituents in the groundwater include zinc ($\leq$ 34 mg/L) and lead ($\leq$ 8 mg/L), which are believed to be infiltrating downward from the surface fill layer, as the concentrations are highest near the water table. Dissolved sulfate concentrations range up to 4000 mg/L and are most concentrated at the base of the aquifer.

The pH of water that passes through the barriers increases from near neutral upgradient to >12 in the BOFS. The high pH conditions result from the dissolution of lime (CaO) and portlandite (Ca(OH)$_2$) contained in the BOFS. As of July 2005, water distinctly influenced by the PRBs (pH=8) migrated to approximately 8 m downgradient of the PRBs, while pH 12 water was at half this distance, indicating moderate retardation of the high pH plume. The pH of water in contact with the BOFS is diminishing gradually as the alkaline materials are depleted and become less exposed possibly due to secondary mineral precipitation. The advance of this high pH water in the aquifer is being monitored.

Dissolved As concentrations decrease sharply from 1-3 mg/L upgradient of the PRB to below detectable values (< 0.002 to 0.02 ppb) in the aquifer sand immediately upgradient of the PRB and within the BOFS. A zone of water with < 0.05 mg/L As has developed between the two barriers and extends ~2.5 m downgradient of the first PRB in July 2005. A similar zone of treated water containing 0.06-0.25 mg/L of As extends ~2.3 m past the downgradient PRB (herein called the second PRB). Monitoring wells situated at ~6.5 m downgradient of the second PRB first showed evidence of treated water (As < 1 mg/L) arriving in July 2005.

A preliminary interpretation of the As treatment mechanisms at the site suggests that dissolved As is
removed from groundwater as it enters the zone where trace amounts of BOFS is present. The removal process begins in the natural materials upgradient of the first detail piezometer bundle (~0.5 m upgradient of the PRB). These upgradient sands accumulated ~150 mg/kg of As (based on whole rock digestion) as of June 2004. Selective extraction results indicate that the As in this zone is probably coprecipitated with secondary amorphous iron hydroxides similar to ferrihydrite (Fe(OH)_3). MINTEQA2 calculations confirm that the water in this zone is slightly supersaturated with respect to ferrihydrite and water analyses confirm that dissolved iron concentrations decrease sharply from > 50 to < 0.1 mg/L in this area. The precipitation of As-bearing iron oxides is induced by the increase in pH conditions in the sand upgradient from the PRB, where trace amounts of BOFS are present. More precise determination of the mechanisms of As removal in this upgradient treatment zone will be completed in the future when more cored material is collected from this area.

The BOFS in the first few centimeters into the PRB had an As concentration ~100 mg/kg above background values, as of June 2004. In these areas, the extraction results suggest that the As is primarily associated with weakly crystalline iron oxide surfaces and aqua regia soluble solids, with lesser amounts that are adsorbed or soluble in weak acid. The nature of the As-bearing phases continues to be investigated. Selective extraction results indicate that As is retained in other ways as well, including association with weak-acid (pH 5) soluble phases and by adsorption to mineral surfaces. The As is also associated with a currently unidentified residual phase that is soluble in aqua regia. The precipitation of discrete As-bearing minerals is not indicated by the MINTEQA2 calculations, although the water is near equilibrium with respect to Ca-arsenate (Ca_3(AsO_4)2·6H_2O) in a few samples from the PRB. Gypsum and calcite have been detected as secondary minerals that are accumulating in the PRB due to the carbonate alkalinity and high sulfate concentrations in the influent groundwater, and the availability of Ca in the slag. The PRB is being monitored for evidence of degraded reactivity toward As due to accumulation of these materials. The precipitation of these secondary minerals may cause a decrease in porosity and hydraulic conductivity in the PRB, but this may be offset by increases in porosity and hydraulic conductivities.

Figure 1: Vertical cross-section of pH and As (mg/L) across the mid-line of the full-scale basic oxygen furnace slag (BOFS) PRB where influent As concentrations are high compared to other parts of the PRB. Data are from three years after installation (2005).
The conductivity of the PRB that occur due to the dissolution of primary minerals in the BOFS.

Dissolved lead and zinc are also removed from the groundwater as it progresses through the PRB. Influent lead concentrations decrease from up to 8 mg/L to below detection (0.05 mg/L) and zinc concentrations decrease from up to 38 mg/L to below detection (0.009 mg/L) in the PRB. The mechanisms of Pb and Zn removal are being evaluated. MINTEQA2 calculations indicate that the pH conditions in Pb and Zn removal zone, the water is near equilibrium to slightly supersaturated with respect to mineral sinks such as Pb(OH)$_2$ and ZnO. The adsorption of heavy metal cations to iron (hydr-oxide) surfaces and the precipitation of hydroxide phases may be responsible for the removal of Pb and Zn. The concentration of As attenuated on the solids was too low on the available core samples to be sufficient for detection by SEM and XRD, however XRF shows distinct enrichment of As (and Zn) on BOFS particle rims.

**Mine Tailings Pilot Scale Iron Filing-OC PRB**

A gold mine tailings impoundment in Canada is the site of a pilot scale PRB installation. At this site, neutral pH tailings porewater contains elevated concentrations of dissolved As. Based on the successful laboratory results of Bain et al. (2003), a homogeneous mixture of zero valent iron filings (Connelly GPM Inc.), locally available wood waste (primarily coniferous tree bark and wood chips) and gravel was installed in an enclosed sheet-piling cell, in a convenient part of the tailings impoundment. The reactive materials were mixed using a standard 10 m$^3$ concrete mixer truck. Enclosing the system isolates it from the ambient tailings, so that geochemical and flow conditions can be controlled. The installation was done in July 2002. The PRB mixture is intended to promote As-sulfide precipitation following bacterially mediated sulfate reduction, in addition to providing an environment where As may be adsorbed or coprecipitated. Locally available organic material was used, rather than attempting to optimize the organic material for sulfate reduction. Adsorption of the dissolved As onto the surfaces of the iron filings and coprecipitation with secondary coatings on the iron are also expected to remove As.

The base of the cell is poured concrete. Because the installation has four walls and a base, flow is constrained to be horizontal through the use of inflow and outflow pumps, located in PRB-wide gravel zones at opposite ends of the PRB. The reactive material forms a unit that is 1.5 m vertical thickness (below the water table), 2 m wide and 3.5 m in the direction of flow (Figure 2). The purpose of this installation is to evaluate the performance of the reactive mixture for the treatment of groundwater As, under controlled flow and As-loading conditions that range from typical to stressed for the site. Neutral pH tailings water containing 20-25 mg/L of As, more than ten times the average groundwater concentration, is continuously pumped into the PRB. Arsenite (AsIII) is the dominant valence of As in the water. No other dissolved metals are at levels of concern. Sulfate concentrations in the water are around 500 mg/L.

Inflow and outflow pumping rates are balanced to maintain linear velocities that range between 2 and 10 times average site groundwater velocities. The PRB was not pumped between December and May most years due to freezing conditions. As of February 2006, more than 75 pore volumes of flow were treated, with residence times ranging from 7 to 14 days. Due to changes in operating conditions at the mine site, water pumping at the PRB was stopped in the spring of 2006.

For more than four seasons of operation, groundwater containing 15-25 mg/L of dissolved As was successfully treated to < 0.01 mg/L in the PRB and as of 2006 there was no evidence of a decrease in reactivity toward As. Testing suggests that the hydraulic conductivity of the mixture remained consistent since the start of the project. Limited precipitation of secondary carbonate minerals may cause a decrease in porosity and hydraulic conductivity, although this may be countered by increases in porosity due to the dissolution of other minerals and organic carbon.
The degree of As removal is consistent throughout the PRB, however, heterogeneities in the flow system through the PRB complicate the interpretation. Groundwater tracer tests using dissolved bromide indicate that water flows three to six times more rapidly through the base of the PRB than in the upper third of the PRB. The tracer information is supported by borehole dilution technique estimates of groundwater velocity. The reasons for the flow heterogeneity are thought to be a result of an increased percentage of gravel and iron in the deeper materials, potentially caused by preferential discharge of the gravel and iron from the concrete mixer truck at the start of PRB infilling and consequently more organic rich material was discharged later in the infilling process. Tracer test information also suggests that preferential flow occurs along the cement-mixture contact. Cores collected from the PRB confirm that the mixture is more gravel and iron rich in the bottom half of the PRB than in the top half.

The pH of water pumped into the PRB rises from 7.4 to 9.5 and the Eh becomes very reduced due to the reduction of water as the iron filings corrode. Recognizing the complexity in the flow system in the PRB cell, the removal of As occurs quickly, with a decrease in As concentrations from ~20 mg/L to < 0.01 mg/L over 1.5 m of travel distance in the fastest flowing part of the PRB. Based on our interpretation of flow in the PRB mixture, this correlates to approximately six days residence time required for this degree of As treatment. In Figure 2, it is evident that elevated As concentrations (> 1 mg/L) occur at several of the points downgradient of where As has already been removed. In these areas, inward seepage of As-bearing groundwater from the tailings adjacent to the PRB is suspected. This is supported by the reoccurrence of other components of untreated tailings water (Ca, Cl, SO₄) in these positions in the PRB, downgradient from where they were at low concentrations. Short-circuiting of flow along the cement base and along the sheet piling walls is also suspected to occur. This allows water to travel from the inflow end of the PRB to the outflow end in less than one day, with minimal contact with the reactive materials.

These specific mechanisms would not be encountered in full-scale applications of the technology because...
a concrete base and sheet piling walls would not be used. Given that the treatment is occurring at As concentrations and groundwater velocities that are higher than are expected at the site, the reactive mixture being used is promising for full-scale application. The PRB is being monitoring for evidence of degraded reactive or hydraulic performance due to accumulation of secondary phases or degradation of the reactive materials.

The mechanisms of As removal are being evaluated. Analyses of cored material indicate that the solids in the deepest parts of the PRB, within 1 m of the input zone are enriched in As by up to 0.76 wt% compared to PRB material that was not exposed to the groundwater. A decrease in dissolved SO$_4$ concentrations, complimented by isotopic enrichment of $^{34}$S in the dissolved SO$_4$, as well as detectable amounts of dissolved H$_2$S support the conclusion that bacterially mediated sulfate reduction is occurring. MINTEQA2 calculations confirm that orpiment (As$_2$S$_3$) is a potential solid phase control on As concentrations. Sequential selective extraction analysis XANES (x-ray absorption near edge spectroscopy) analyses (Brookhaven National Lab, beamline X15B and Argonne National Lab GSECARS) and mineralogy are being done to gain insight into the As removal mechanisms. Preliminary analysis of the extraction data suggest that the As in the reactive materials is primarily (>50% of the total As) released under aggressive extraction steps (aqua regia) and the remaining As is divided among the less aggressive extraction steps (potassium phosphate, sodium acetate and oxalic acid based extractions). This information suggests that most of the As is associated with crystalline phases (possible As sulfides) and less is adsorbed or coprecipitated with iron oxyhydroxides. The XRF and XANES analyses suggest that As and S are frequently associated on the PRB solids, and often are in a reduced state. A mineralogical examination indicates that As is associated (adsorbed and coprecipitated) with Fe oxyhydroxides that have replaced Fe in the filings and with Fe oxyhydroxides that have rimmed and partly replaced the exterior portions of the organic matter. Sulfide minerals were not detected in the mineralogical examination, perhaps due to their amorphous character.

**Phosphate fertilizer site, pilot scale iron filing-limestone-OC PRB**

Water at this site differs distinctly from the other two sites. Here, sulfuric acid was used in the production of fertilizer. The groundwater at the site has low pH values (2-4) and high concentrations of dissolved As (up to several hundred mg/L at the base of the plume), as well as elevated concentrations of Fe, Cd and Pb. A reactive mixture consisting of zero valent iron filings, compost and gravel was installed in an excavated trench. Five percent by volume limestone gravel was added to the mixture to improve pH conditions for sulfate reducing bacteria. The trench was held open during construction using guar gum biopolymer, which was displaced out of the trench during placement of the reactive mixture. The pilot scale PRB is 8.2 m wide (across flow), extends 2.3 m below the water table and is 1.8 m in the direction of flow (Figure 3). The PRB was installed in the fall of 2002 and has been monitored by UW and the U.S. EPA. In this document we will highlight how the reactive materials perform in comparison to the pilot scale PRB at the gold mine.

This pilot scale PRB has been operating for more than three years. This PRB is similar in composition to the one described for the gold mine, except that it contains limestone to neutralize the acidity of the groundwater entering the PRB, to increase the potential for bacterially mediated sulfate reduction to occur. Sulfate reduction brings with it the potential to remove dissolved As, Fe and heavy metals (Pb, Cd) from the groundwater in the form of sulfide minerals. The removal of Fe(II) from solution will reduce the acid-generating potential of this water in the receiving water body, while the alkalinity generated during sulfate reduction will improve the low pH conditions prevalent at this site.

The PRB is situated in a part of the property where groundwater flow is very slow and the residence time for porewater in the PRB is approximately 5 months. Approximately 6 pore volumes of water have gone through the PRB as of February 2006. Upon entering the PRB, groundwater As concentrations decrease
from values ≤ 126 mg/L to < 0.04 mg/L. The concentration of dissolved Pb ~3 mg/L to < 0.01 mg/L and dissolved Cd concentrations decrease from ~2 mg/L to < 0.003 mg/L. As was noted in the gold mine pilot scale PRB, decreased SO₄ concentrations, the presence of dissolved H₂S and enriched ³⁴S values in the sulfate and a viable population of sulfate reducing bacteria support the interpretation that sulfate reduction followed by metal sulfide precipitation is occurring and could be responsible for the removal of metals from the water. The alkalinity also increases significantly, but evidence that this is a result of sulfate reduction rather than limestone dissolution is not currently available. A plume of clean water is starting to develop downgradient of the PRB, although evidence of this progress is complicated by high concentrations of metals downgradient of the PRB and slow groundwater velocities. Ongoing monitoring at this site is being conducted by the U.S. EPA.

**FIGURE 3.** Vertical cross-section of pH, As and Cd within the pilot scale iron filings + organic carbon + limestone PRB installed at the fertilizer manufacturing facility. Data are from 18 months after installation.

**Power Distribution Site BOFS PRB**

An electrical distribution station in Ontario, Canada is the site of a BOFS PRB system. The system was installed in the fall of 2002. Groundwater containing up to 0.4 mg/L As is directed into a concrete
chamber that contains 2 m³ of a 50% BOFS, 50% gravel mix. Other dissolved components of the water are at dilute levels. The residence time for water in this chamber is approximately one day. Within the chamber, dissolved As concentrations decrease to < 5 ppb. Due to the high lime/Portlandite content of the BOFS, the pH of effluent is near 12. To manage the high pH effluent, a 20 m² collection bed containing a ~75 cm layer of peat, wood chips and gravel was constructed. Laboratory batch testing suggested that peat would be an effective remediation for the high pH water because the natural decay of peat releases organic acids (e.g. humic and fulvic acids). Effluent from the BOFS chamber is distributed through PVC tile drain into the peat layer. The drainage is collected in a continuous polyethylene liner that underlies the peat. Since it was installed in Sept. 2003, the peat layer has been effective in lowering the pH to between 6 and 7. Monitoring at this site is being conducted by Ontario Power Generation.

Summary

The effective removal of As in groundwater under highly different geochemical conditions attests to the viability of mixtures based on zero valent iron and organic carbon and also with basic oxygen furnace slag. Both As(III) and As(V) in groundwater are effectively treated. In the iron filing + OC PRBs there is supporting evidence of the precipitation of sulfide minerals and coprecipitation of As with iron oxyhydroxides on the iron filings and with iron oxyhydroxides that occur as coatings on organic carbon, as primary mechanisms of As removal. In the mine site PRB, complete removal of As from high concentrations (averaging 20 mg/L) has been observed for more than four years and more than 70 PV of flow. The PRB operating conditions are more extreme (higher groundwater velocity and higher concentrations) than would be encountered in a full-scale PRB installation at the site. At the fertilizer site, acidic pH conditions in the incoming water are neutralized by limestone that was included in the mixture. As a result, conditions favourable for the removal of As, Cd and Pb are generated inside of the PRB. Low groundwater flow velocities in the PRB location allow very high concentrations of dissolved As to be removed from the groundwater. Additional efforts are under way to determine the significance of As removal by adsorption onto and coprecipitation with secondary phases that probably are forming on the surfaces of the zero valent iron. The two BOFS installatons, one with high concentrations of dissolved As, Pb, Zn, and SO₄ and the other with only As at 0.4 mg/L as the concern demonstrate complete removal of As from the water for more than four years. In the latter case, the high pH effluent was managed using a peat drain; in the former, observations of the conditions are continuing. In all of the PRBs, there is no evidence to date for degrading hydraulic properties or As-removing ability, but observations for this are continuing.

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