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TECHNICAL ARTICLE

Biogeochemical Analysis of Spent Media from a 15-Year Old Passive Treatment System Vertical Flow Bioreactor

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Abstract
Spent organic media from the vertical flow bioreactor process unit of a passive treatment system (PTS), which received acidic drainage from an abandoned coal mine for 15 years, was evaluated. The Jennings PTS (Butler County, Pennsylvania, USA) was built in 1997. The vertical flow bioreactor was one of the first constructed in the late 1990s and included a mixed spent mushroom compost (272 t) and limestone aggregate (345 t) media. The media was expected to effectively function for 14 years. After 15 years of operation, hydraulic conductivity problems resulted in its removal and replacement. Samples of the spent media were analyzed to determine total and leachable [by toxicity characteristic leaching procedure (TCLP) extraction] metal concentrations. Remaining organic matter was estimated by combustion as loss-on-ignition. Spent media total metals analyses yielded mean concentrations of 72 g/kg Fe, 37 g/kg Al, and 0.64 g/kg Mn. The TCLP results indicated that concentrations of As, Cd, Cr, and Pb were far below USA Resource Conservation and Recovery Act regulatory criteria. Total metal concentrations in the spent media were less than the mass load removals calculated, based on the water quality and quantity data.

Keywords Organic substrate · Bacterial sulfate reduction · Leachable metals · RCRA

Introduction

Passive treatment systems (PTS) typically consist of a series of ecologically-engineered pond- and wetland-type process units that promote specific physicochemical, biogeochemical and microbiological processes to treat metal-contaminated mine water (Hedin et al. 1994; Nairn et al. 2009; Skousen et al. 2017; Watzlaf et al. 2004; Zipper and Skousen 2010; Zipper et al. 2011). Specific process unit designs, sizing, and sequences depend on site-specific untreated water quality and flow assessments.

Vertical flow bioreactors (VFBRs), often referred to as vertical flow ponds or vertical flow wetlands, rely on sulfate-reducing bacteria to oxidize exogenous organic matter and reduce sulfate to sulfide, thus precipitating metals as metal sulfides (Das et al. 2012; Hedin et al. 1994; Nairn et al. 2009; Skousen et al. 2017; Taylor et al. 2005; Watzlaf et al. 2004). In addition, these systems often contain limestone for acid neutralization. VFBRs have been demonstrated to be feasible treatment alternatives for acid mine drainage (AMD) due to reliable metal removal, precipitant stability, minimal energy consumption, and low operational costs (Neculita et al. 2007; Watzlaf et al. 2004; Zipper et al. 2011). Once the VFBR substrate has reached the end of its operational life, management of the organic, metal-laden media can have significant excavation and disposal costs, especially if the material is classified as a hazardous waste.

Organic substrates vary significantly, depending on location, and are often selected based on local availability. Organic media options, individually or in various combinations, can include spent mushroom compost, wood chips, saw dust, rice straw, and cow manure (Das et al. 2012; Nairn et al. 2009; Song et al. 2012). Many organic substrate evaluation studies were relatively short-term bench-scale experiments and, although results varied, trace metal removal was documented, regardless of organic substrate (Bhattacharya

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et al. 2008; Cheong et al. 2010; Das et al. 2012; Jarvis et al. 2014; LaBar and Nairn 2016, 2018; Nairn et al. 2009; Song et al. 2012; Younger et al. 2002). In many full-scale systems, media mass or volumes are calculated to last about 20 years. However, due to decreased hydraulic conductivity over time, full-scale process units rarely function as designed for the full 20 years without significant maintenance (Denholm et al. 2010; Neculita et al. 2007; Skousen et al. 2017; Watzlaf et al. 2004), though a lack of well-documented monitoring makes it difficult to identify specific causes for performance declines. Neculita et al. (2007) and Watzlaf et al. (2004) noted that permeability problems are often associated with the accumulation of iron oxyhydroxide precipitation on the surface of organic substrates, metal sulfide accumulation within the organic layer, and compaction of the organic substrates. Denholm et al. (2010) found stirring media with hydraulic conductivity issues reestablished flow and extended the life of the media.

When VFBR performance declines or fails, organic substrates are removed and replaced. Spent substrates must be handled in an environmentally appropriate manner. In the USA, toxicity is typically evaluated using the toxicity characteristic leaching procedure (TCLP; USEPA SW-846 method 1311), which simulates leaching in conventional landfill materials. If the results indicate metals concentrations greater than Resource Conservation and Recovery Act (RCRA) criteria, the material is considered hazardous. The objectives of this study were to analyze spent media from a 15 years old VFBR for total and TCLP-leachable metal concentrations, remaining organic matter, and the total accumulated mass of metals, based on the remaining mass of organic material.

Methods

Site Description

The Jennings PTS, located at the Jennings Environmental Education Center in Butler County, Pennsylvania, was built in 1997 to treat acidic, metal-laden drainage from an abandoned coal mine. The untreated water contained approximately 270 mg/L total acidity, 33 mg/L total Fe, 14 mg/L total Mn, and 14 mg/L total Al, at pH 3.1. Volumetric inflow discharge rates were approximately 75 L/min (Denholm et al. 2010; Rose 2004; Rose and Dietz 2002; Skousen et al. 2017; Watzlaf et al. 1994). The Jennings PTS included a VFBR, a combined aerobic wetland/settling pond, and final aerobic wetlands (Fig. 1). It should be noted that the untreated AMD flows directly into the VFBR.

The VFBR included an underdrain network of 2 cm perforated pipes, spaced 1.8 m apart and placed in 30 cm of AASHTO #57 non-reactive river gravel covered by woven geotextile (Fig. 1). The overlying media consisted of 272 tonnes (t) of spent mushroom compost mixed with 345 t of limestone aggregate (Denholm et al. 2010). Using an estimated media porosity of 0.5, the hydraulic retention time in the media layer of the VFBR was $\approx$ 36 h. The mushroom compost placed into the VFBR had an estimated moisture content (MC) and organic matter (OM) content of 62% and 26%, respectively (Hy-Tech Mushroom Compost Inc. 2015). The MC and OM values were obtained from the mushroom farm before the compost was used to grow mushrooms, and were the best approximation based on available data.

Initial calculations indicated that the VFBR was expected to perform for a decade, with a maximum lifetime of 14 years. After 7 years of operation, decreased hydraulic conductivity was noted. The hydraulic conductivity was considered to be compromised when the water cap on the VFBR began to discharge over the emergency spillway, approximately 0.3–0.5 m above the designed water elevation (Fig. 1). The VFBR was drained and the media was stirred in 2004 using heavy machinery to restore the hydraulic conductivity. The stirring reestablished the hydraulic conductivity for a few years, but two additional stirring events were necessary in 2007 and 2011. Each stirring resulted in a month of system downtime, with most of the time spent draining the pond (Denholm et al. 2010). The odor of hydrogen sulfide was noted during the mixing events but nothing that was considered out of the ordinary for a VFBR during the summer months. During the two stirring events in 2004 and 2007, the geotextile liner was not damaged, though protecting the geotextile liner potentially limited the effectiveness of the stirring events and might have allowed fines to settle on the liner. During the 2011 stirring event, a filter cake layer was observed accumulating on the liner. The caking was assumed to be contributing to the loss of hydraulic conductivity, and the geotextile liner was removed during the 2011 stirring event. However, after 15 years of operation in 2012, and failure to reestablish appropriate hydraulic conductivity, the spent media was removed, encapsulated on site, and replaced by new treatment media. Samples of the spent media were collected and are the subject of this study.

Sample Collection and Analysis

Six samples were collected in 2012 from the excavated bulk media and placed into individual 3.8 L low density polyethylene bags. Although it was not feasible to fully homogenize the bulk media prior to sampling due to the amount of material, best efforts were made, and the grab samples taken were felt to be as representative as possible. Samples were maintained at 4 °C until analysis. All samples were analyzed using standard methods (Fig. 2, Nelson and Sommers 1996; USEPA 2014) in the Center for Restoration of Ecosystems and Watersheds (CREW) laboratories at the University

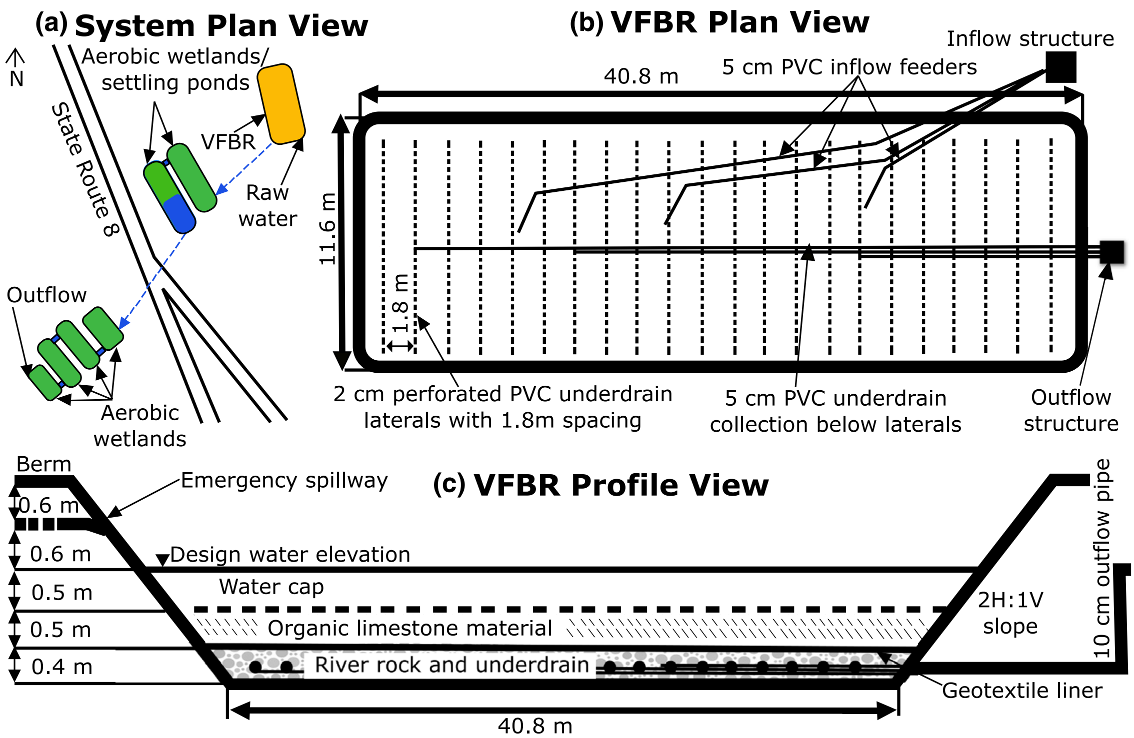

Fig. 1 **a** Plan view of Jennings passive treatment system located at the Jennings Environmental Education Center in Butler County, Pennsylvania; **b** plan view of the vertical flow bioreactor (VFBR) process unit and **c** profile view of the VFBR process unit

of Oklahoma. Representative aliquots for analyses excluded large pieces of limestone. No evidence of armoring or metal co-precipitation on limestone was noted, and accumulated metals were found predominantly in the organic substrate (LaBar and Nairn 2017, 2018). Spent media aliquots for organic matter estimation by loss-on-ignition (LOI) were crushed using a mortar and pestle and passed through a #60 (< 250 µm) sieve. Visual inspection indicated that smaller limestone fragments were retained while organic material passed through the sieve. Substrate aliquots were oven dried at 105 °C for 24 h and consistent weight was achieved to determine moisture content by gravimetric difference. Dried samples were then combusted in a Sybron ThermoLyne muffle furnace at 550 °C for 4 h to determine organic matter as LOI by gravimetric difference. These same samples were then re-combusted at 550 °C for 16 h, resulting in an overall change of less than 1% by mass. The initial OM values (determined at 550 °C for 4 h) were used for the calculations to ensure that loss of inorganic components did not erroneously contribute to OM estimates.

Results and Discussion

Water Quality

Over the 15 years sampling period, water quality samples were periodically collected at the inflow and outflow of the VFBR (Stream Restoration Inc. 2015). The annual average water quality in and out of the VFBR indicated that the Jennings PTS demonstrated significant Al and Fe removal (one-tailed paired student *t* test, t(df) = 13, $p < 0.05$). During the 15 years lifetime of the VFBR media, the pH increased on average from 3.1 to 6.6, likely due to bicarbonate generation via both limestone dissolution and sulfate reduction. Aqueous total Al and Fe concentrations decreased by 90% and 60%, respectively (Table 1). Given the amphoteric nature of its solubility, Al retention likely occurred due to precipitation as $Al(OH)_3$ solids, and through exchange and sorption (LaBar and Nairn 2017, 2018). Fe retention was likely a result of a combination of processes. Because the Jennings VFBR was the first process unit in the treatment system, precipitation of FeOOH solids was noted on the surface of the substrate. Sulfate reduction also likely contributed to Fe removal through formation of FeS and FeS_2 precipitates. In addition, exchange and sorption processes in the organic media likely contributed to Fe retention (LaBar and Nairn 2018). Aqueous Mn concentration changes were minimal in

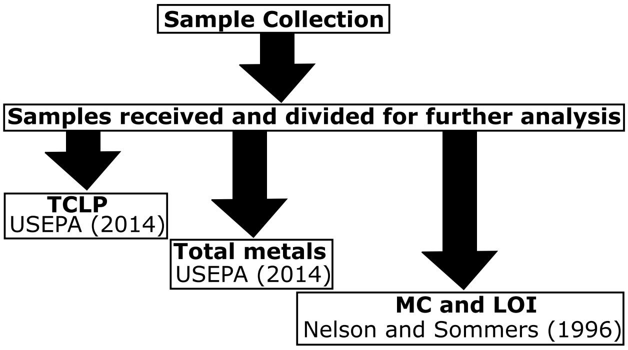

Fig. 2 Flowchart of methods used to analyze spent media from the vertical flow bioreactor, including Toxicity Characteristic Leaching Procedure (TCLP), total recoverable metals, moisture content (MC), and organic matter as loss on ignition (LOI)

the VFBR, with a 2.5% mean decrease (Table 1). Manganese was likely retained via sorption, exchange, and carbonate precipitation (LaBar and Nairn 2018). These results were similar to those published by Hedin et al. (2013), which found that vertical flow ponds effectively retained Al and partially retained Fe but had minimal effect on Mn concentrations. Due to inaccurate and inconsistent methods, the sulfate data before 2007 was removed because it did not meet the quality assurance/quality control standards of this study. The sulfate concentrations post-2007 support sulfate reduction during the remainder of the media life, which included two of the stirring events.

Total Metals in Spent Media

Fe, Ca, and Al accounted for most of the metals found in the spent media. Concentrations of Al and Ca each accounted for more than 3% (> 30 g/kg) of the total sample by mass, with Fe concentrations exceeding 6% (> 66 mg/kg, Table 2). These elevated solids concentrations were expected due to the significant decreases in Al and Fe aqueous concentrations over the lifespan of the VFBR media. Elevated Ca was partially attributed to the initial Ca concentration in the spent media (1.8%) (Hy-Tech Mushroom Compost Inc. 2015). The remaining Ca and Mg concentrations in the media are due to the dissolution of limestone in the organic substrate by the low pH water. Although bulk influent and effluent water quality data did not reflect substantial Mn retention, mass loading resulted in measurable substrate concentrations, attributed in part to impurities in the limestone. These results were similar to those published by Hedin et al. (2013), which found vertical flow ponds were effective at removal of Al and partial removal of Fe but had minimal effect on Mn.

Other metals that were not targeted for treatment were rarely measured in regular water quality monitoring. However, after 15 years of operation, the VFBR substrate contained numerous other metals including Zn, Cd, Cr, and Pb. The elevated concentrations of Zn in the spent media suggest there was Zn removal from the AMD, which has been reported in numerous studies (Gandy and Jarvis 2012; Jarvis et al. 2014, 2015; Nairn et al. 2009; Younger et al. 2002). However, Zn mass loads could not be calculated due to the lack of Zn water quality data.

TCLP of Metals in Spent Media

Metals concentrations in TCLP extracts were two orders of magnitude less than applicable RCRA standards. It must be noted that only four (As, Cd, Cr, and Pb) of the eight RCRA metals were analyzed as the others (Ba, Hg, Se, and Ag) were not expected to be present in the waters being treated and reliable analytical capabilities were not fully developed in the laboratory at the time of analysis. The excavated spent media sequestered the measured trace metals, but they were not readily leachable via the USEPA-approved TCLP (Table 3). Therefore, it was determined that the spent media likely did not require disposal at a permitted hazardous waste facility.

Estimation of Total Metals Removed

The total mass of metals retained in the spent media was empirically compared using available water quality data and the substrate total metals concentrations. Metal retention was estimated using the mean volumetric discharge rate into the VFBR of 75.7 L/min and an outflow rate of 72.5 L/min, operating for 15 years and the available water quality data, incorporating three months down time for known maintenance events (Table 4). The organic content was used to determine the approximate mass of the material excavated.

Table 1 Jennings vertical flow bioreactor (VFBR) mean changes in pH, flow, and total Al, Fe, and Mn concentrations over a 15 years period, and median sulfate concentrations over a 5 years period (Stream Restoration Inc. 2015)

	Sample size	pH	Flow (L/min)	Al (mg/L)	Fe (mg/L)	Mn (mg/L)	Sulfate* (mg/L)
VFBR influent	108	3.1	75.7	13.6	33.7	13.7	738
VFBR effluent	124	6.6	72.5	1.7	12.7	13.5	701

*Sulfate samples were analyzed from 2007 to 2012, with a sample size of eight for each location

Table 2 Total metals concentrations (g/kg) for six samples of Jennings VFBR spent media after 15 years of operation

	Al	Ca	Cd	Co	Cr	Cu	Fe	Mg	Mn	Ni	Pb	Zn
Mean	37.0	40.7	0.010	0.279	0.025	0.067	72.1	1.68	0.647	0.646	0.043	1.199
Standard dev.	1.69	1.78	0.001	0.014	0.001	0.002	3.63	0.34	0.034	0.032	0.001	0.051
Minimum	34.2	38.2	0.010	0.254	0.023	0.062	66.2	1.36	0.584	0.590	0.041	1.114
Maximum	39.1	43.5	0.011	0.302	0.026	0.070	76.3	2.42	0.685	0.695	0.045	1.260

Table 3 Selected leachable metals concentrations (mg/L) in TCLP extracts of Jennings VFBR spent media

	As	Cd	Cr	Pb
RCRA criteria	5	1	5	5
Mean	< 0.02[a]	0.006	0.002	0.029
Standard dev.	–	0.0007	0.0013	0.0031
Maximum	–	0.007	0.004	0.034
Minimum	–	0.006	0.001	0.026

[a]Arsenic concentrations were below instrument detection limits for all samples

Table 4 Mass of accumulated metals retained (in kg) in the Jennings VFBR based on influent and effluent water quality and selected total metals concentrations of the spent media

	Water quality	Spent media
Al	7030	2300
Cd		0.62
Cr		1.56
Fe	12,240	4490
Mn	474	40.3
Pb		2.68
Zn		74.6

Since the limestone was separated during the analyses, only the mass of the mushroom compost was considered for these calculations. The initial OM content of 25.9% includes the 62% MC of the media. Therefore the 272 t of initial media includes 196 t of water and 103 t of solids. This results in a dry OM content of 68.4% (71 t), per Eq. (1).

$$\frac{25.9\%\ OM\ wet\ mass}{(1 - 62.0\%\ MC)} = 68.4\%\ OM\ dry\ mass \tag{1}$$

The LOI analyses showed that 25.6% of the OM remained in the spent media, Therefore, over the 15 years lifespan of the media, the OM had decreased from 68.4% to 25.6% ± 0.79 (Table 5). The Ca concentration measured in the media was attributed to the limestone in the mixture and was converted to $CaCO_3$ for all calculations. The total metals concentrations were summed and converted to kg/kg to represent percent metals of the media, which was approximately 21.5%. The initial non-OM solids mass, 32.9 t, was assumed to remain constant throughout the lifetime of the system, and the only loss of mass was assumed to be OM. The OM and total metals percentages were then used to determine the total mass of solids excavated from the VFBR. The total mass of solids remaining in 2012 was then calculated to be 62.3 t. Using the total mass of solids, the masses of each metal were calculated from the percentages (Eq. (2) and Tables 4 and 5).

$$\frac{32.9\,\text{t}\ initial\ non\text{-}OM\ solids}{1 - (0.215\,metals + 0.256\,OM)} = 62.3\,\text{t}\ dry\ solids \tag{2}$$

Discrepancies between the estimates of media metal concentrations based on water quality mass removal and solids analyses of the organic media for the lifetime of the system were expected. The mass removal calculated using water quality concentrations and flow data is approximately three times the mass removal estimated from the spent media analyses. It is important to note that the solids data only represent the mass of metals retained within the organic media layer. The unaccounted mass of metals likely precipitated at the surface of the media, in the caking layer accumulated on the geotextile, and in the river gravel underdrain. Although best efforts were made to collect representative samples, it would be difficult and unrealistic to seamlessly homogenize over 100 t of material using an excavator. Therefore, metal mass precipitated at the surface of the media and in the caking layer accumulated on the geotextile, which likely represents a substantial fraction of total removal, may not be accurately represented in the samples collected. Additionally, it was assumed that the material analyzed represented only the remaining spent mushroom substrate placed 15 years earlier and that no other materials were purposely or inadvertently added. The role of materials that may have eroded into the system or the inclusion of volunteer vegetation was not evaluated. Any alteration to the total mass of spent media could have substantially influenced the resulting calculations.

Although the remaining 16 t of OM indicate that the VFBR had the potential for continued biological AMD treatment, the VFBR physically failed due to decreased hydraulic conductivity. This demonstrates the importance of selecting a combination of materials that not only meets the biological criteria but maintains hydraulic conductivity

Table 5 Changes in mushroom compost composition after 15 years of use in the Jennings VFBR

	1997	2012
Mass of media (wet) (t)	272	
Moisture content (%)	62.0	
Mass of solids (dry) (t)	103.4	62.3
Organic matter content (%)	68.4	25.6
Mass of organic matter (t)	70.5	15.9
Mass of non-organic matter (t)	32.9	32.9
Mass of metals in compost (t)		13.4

for the designed lifespan of the VFBR. The decrease in hydraulic conductivity of the VFBR was likely the result of compaction of the organic layer and accumulation of precipitates from the elevated concentrations of Al and Fe entering the VFBR (Neculita et al. 2007; Watzlaf et al. 2004). Flow paths through the media could have been obstructed by accumulated precipitates, particularly at the surface of the media (Watzlaf et al. 2004). Stirring events likely created and recreated flow paths by redistributing the accumulated metals in the top of the media layer for a short period of time. The media showed a more rapid decrease in hydraulic function after each stirring event, with the first event extending function for 4 years, but by the third stirring event, the benefit lasted less than a year, resulting in the decision to replace the media (Denholm et al. 2010).

To aid in further work, depth-incremented sampling of the media before stirring and sampling of the river gravel underdrain may help address the differences in the estimates of media metal concentrations based on water quality and solids analyses. Similar analysis of additional passive treatment systems containing VFBRs, which treat mine drainage sources with a varied water quality, would also assist in validating the potential importance of initial process units in removal of metals present at elevated concentrations.

Conclusions

The Jennings VFBR was effective at increasing pH and removing Al and Fe from AMD with no pretreatment for 15 years. However, decreased hydraulic conductivity in the VFBR necessitated replacement of the media, despite three stirring events spanning an 8 years period. A substantial mass of metals accumulated in the media and were sequestered, but they were not readily leachable. For those metals analyzed, leachable concentrations in the spent media from the Jennings VFBR did not exceed RCRA criteria. The media had a quarter of its original OM and therefore was likely biologically capable of providing additional years of AMD treatment. This research highlights the importance of proper operation and maintenance of VFBRs. Notably, decreased hydraulic conductivity may not be an indicator that the media has reached its treatment capacity. Performing regular periodic site visits, with the expectation that stirring may be necessary to utilize the system's full potential before requiring a complete overhaul, are critical to long-term system evaluation (Denholm et al. 2010).

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