LABORATORY AND PILOT-SCALE STUDIES ON THE TREATMENT OF ACID ROCK DRAINAGE AT A CLOSED GOLD-MINING OPERATION IN CALIFORNIA[1]

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Abstract: Acid rock drainage from a closed gold-mining operation in northern California was studied first in the laboratory and then on the pilot scale to determine the technical feasibility of passive treatment. The drainage has a pH of 3.8, and concentrations of Cu, Fe, Mn, Ni, and Zn of 140, 190, 28, 0.93, and 40 mg/L respectively. The laboratory studies concentrated on the question of whether local organic and soil materials could be used to support sulfate reduction in a passive treatment system. Samples were incubated at laboratory temperatures for a period of 4 weeks. Soil and wood processing wastes from the immediate vicinity proved to be too acidic to maintain a large population of sulfate reducers. The most reasonable material for sulfate reduction was a mixture of equal amounts by weight of cow manure, planter mix soil, and limestone chips. The final solutions had pH's of 6.5 to 6.9, and average Cu, Fe, Mn, Ni, and Zn concentrations of 0.02, 1, 5, 0.05, and 0.1 mg/L, respectively. Based on the laboratory results, a pilot system was constructed that consisted of a lined steel container filled with a substrate volume that measured 2 by 3 by 12 m. The substrate mixture was the same as used in the laboratory tests. Raw manure from a dairy farm was mixed into the substrate for the sulfate-reducing bacterial (SRB) inoculum. Loading of the system was based on the estimate that 0.3 mol sulfide per cubic meter of substrate per day would be generated, and the inflow of heavy metals should not exceed the sulfide generated. Using these principles, the flow was set at approximately 800 mL/min. Over the course of 9 months, the pilot system achieved removal of Cu and Ni below the effluent standards of 1.0 and 0.7 mg/L. Dissolved Zn concentrations in the effluent averaged approximately 0.1 mg/L, compared with an effluent standard of 0.02 mg/L. Dissolved Fe concentrations in the effluent varied with the seasons, reaching a minimum of 1 mg/L in the summer and rising to a maximum of 120 mg/L in the winter. There is a significant increase in concentrations of Fe in unfiltered waters. This implies that, in a full-scale system, a settling-polishing pond will be needed.

Additional Key Words: Constructed wetland, heavy metals treatment, sulfate reduction, pilot scale reactors

Introduction

In 1989, the use of anaerobic, subsurface wetlands that emphasize sulfate-reduction was in the developmental stage. In eastern United States, systems using mushroom compost that treated coal mine drainage were being studied by scientists at the U. S. Bureau of Mines (Hedin et al. 1988 and 1989, McIntire and Edenborn 1990). In the West, research at the Colorado School of Mines (CSM) was concentrating on the removal of contaminants from metal-mine drainages by sulfate reduction, also using mushroom compost as the substrate (Machemer and Wildeman 1992, Machemer et al. 1993, Wildeman et al. 1993). In 1990, two breakthroughs on wetlands treatment significantly advanced research at CSM: (1) The determination that the primary anaerobic removal process is sulfate reduction and sulfide precipitation mediated by microbes (Reynolds et al. 1991). (2) The loading of a sulfate-reducing system can be determined by the rule of thumb that the level of microbial sulfate reduction should always exceed the amount of sulfide precipitation (Machemer et al. 1993, Wildeman et al. 1993).

These two findings allowed development of anaerobic wetland systems by a staged design process similar to other mineral processing schemes. In particular, because microbial processes dominated, laboratory studies to determine suitable substrates and sulfate-reducing inocula can be conducted with a reasonable degree of certainty. Also, these laboratory studies could be conducted to determine if "in principle" a treatment technique can be effective. Then, because effective treatment relies upon a balance between sulfate reduction and metal sulfide precipitation, bench- and pilot-scale experiments can determine the proper loading factors and how a treatment system can be adapted to on-site conditions.

This paper reports on the first pilot-scale passive treatment system that was constructed using these concepts. The site was the Grey Eagle gold mine in northern California, where it is necessary to treat a continuous flow of seepage from a tailings impoundment. The seepage also contains acid rock drainage from historic mining. The composition of the commingled waste water stream is given in table 3. The mine site is owned by Noranda Grey Eagle Mines Inc. which funded the development project.

Laboratory Study

The laboratory study concentrated on microbiology and had the following objectives: (1) To identify a suitable substrate of soil and organic material that would be readily available at the mine site. (2) To measure the removal of the contaminants from the discharge water by analyzing the changes in the substrate-water system with time. (3) To determine a local source of SRB that can be used as the inoculum for the pilot cell.

Local soils and wood wastes were collected from the vicinity of the mine, and were mixed with water in a 1:1 by volume ratio. These paste samples were analyzed for pH, Eh, and electrical conductivity. These general tests indicated that the most easily available materials were unsuitable, primarily because the paste pH's were less than 5. At this low pH, sulfate reduction is severely inhibited (Postgate 1979). To meet objective 2 and determine what types of materials could be considered for objective 1, a protocol for microbiological tests was devised as shown in table 1. It was assumed that limestone, cow manure, and organic soil could be found close to the mine site, so this was established as the base substrate. Other substrates used at the Big Five pilot system in Idaho Springs, CO (Wildeman et al. 1993) were used to give a more complete test of objective 2. To test objective 3, various materials were used without a sulfate-reducing bacterial inoculum being added.

Two features of this type of laboratory study help with the success of the final treatment design. First, qualitative observations and indicative measurements are just as important as analytical measurements. Consequently, during the 4-week study, pH, Eh, color, and odor within the bottles were measured. Second, because the analytical tests are simple, a large number of combinations of organic material, soil, and other amendments can be investigated. At the end of the experiment, selected bottles were sent for analysis in which filtered samples of the supernatant water were analyzed for a suite of metals.

Table 1. Experimental protocol for the laboratory tests on the mine drainage.

Base substrate: 1/3 planter soil mix, 1/3 manure, 1/3 limestone chips.
Base mix: 30 g of substrate, 90 g of mine drainage. For the anaerobic cultures, add a minor amount of distilled and sterilized water to fill to the top of the bottle. For the aerobic cultures, no extra water is added and the bottle is left 3/4 full. Record the extra water added.

Bottle No.	Culture Bottle Composition
1 - 6	Base substrate with base drainage-substrate mix.
7 - 9	Big Five Cell E substrate with base drainage -substrate mix.
10	Base substrate with mix of 10 g substrate to 100 mL drainage.
11	Base substrate with mix of 25 g substrate to 100 mL drainage.
12	Base substrate with mix of 60 g substrate to 60 mL drainage.
13	Cow manure with base drainage-substrate mix.
14	Sheep manure with base drainage-substrate mix.
15	Planter mix with base drainage-substrate mix.
16	Limestone chips with base drainage-substrate mix.
17	1/4 planter mix, 3/4 manure with base drainage-substrate mix.
18	1/2 limestone chips, 1/2 manure with base drainage-substrate mix.
19	1/2 planter mix, 1/2 manure with base drainage-substrate mix.
20	1/3 limestone, 1/3 sawdust, 1/3 manure with base drainage-substrate mix.
21	1/3 limestone, 1/3 sawdust, 1/3 planter mix with base drainage-substrate mix.
22	1/3 planter mix, 1/3 sawdust, 1/3 manure mix with base drainage - substrate mix.
23	Original Big Five mushroom compost with base drainage-substrate mix.
24-35	Bottles 1 - 5, 8, 17, 19, 20, 21, 22 and 23 run under aerobic conditions.
36	Base drainage-substrate mix with 10 g of Big Five Cell E substrate added.

Table 2. For the test bottles in table 1, visual observations, pH, electrochemical measurements of hydrogen sulfide, and colorimetric measurements of dissolved copper.

Bottle	Color[1]	pH	H_2S[2]	Copper[2]
1	B	6.5	+	±
2	B	6.7	+	±
3	B	6.7	+	0
4	B	6,7	+	0
5	B	6.7	+	0
6	S	6.8	+	0
7	SS	5,8	+	+
8	SS	5.7	+	NA
9	SS	5.6	0	NA
10	A	6.2	0	NA
11	B	6.7	+	NA
12	B	6.9	+	NA
13	B	6.8	+	NA
14	S	7.1	0	NA
15	A	6.5	0	NA
16	A	6.1	0	0
17	B	7.0	+	NA
18	B	6.9	+	NA
19	B	6.9	+	NA
20	A	6.1	0	NA
21	A	4.7	0	NA
22	S	5.9	0	NA
23	S	6.3	0	NA
24	SS	7.0	0	NA
25	SS	6.9	0	NA
26	SS	6.9	0	NA
27	SS	6.9	0	NA
28	SS	6.8	0	NA
29	SS	4.0	0	NA
30	B	7.0	0	NA
31	B	7.8	0	NA
32	S	7.4	0	NA
33	A	3.9	0	NA
34	A	6.8	0	NA
35	A	6.2	0	NA
36	B	6.9	+	NA

[1]B = Bottle black throughout; SS = all of sediment blackened; S = part of sediment blackened; A = no blackening, water is clear or pale brown.
[2]For the hydrogen sulfide and copper tests, qualitative estimates were made and ++ = strongly positive, + = positive, ± = uncertain, and 0 = not detected, and NA = not analyzed.

Table 2 gives the 2-week results of indicative observations on the bottles. Already strong indications of what substrates would successfully promote the growth of sulfate-reducing bacteria are seen. For example, in bottles 17 and 19, which had appreciable manure in the substrate, the bottles had blackened and H_2S was present. Bottles 30 and 31 had the same substrate mixes as bottles 17 and 19, but the caps had been loosened in an attempt to keep the bottles aerobic. However, sulfate reducing bacterial activity was so strong that these bottles had also turned black. Bottle 21 with no manure had a low pH and no blackening.

The results of analyses on selected bottles are shown in table 3. Although this is a static experiment, the concentrations of contaminants give some indication of what to expect in a flowing system. First, concentrations of sulfate show that vigorous sulfate reduction occurred in bottles (1, 9, 10, 11, 12, 36) that contained organic material other than mushroom compost. Next, in the bottles that contained varying amounts of the base substrate of 1/3

Table 3. Contaminant concentrations in mg/L in the original drainage water (OW), the final supernatant waters in the test bottles, and the regulatory effluent limit (EF).

Bottle or Sample	Cd	Cu	Fe	Mn	Ni	Zn	$SO_4=$
OW	0.088	140	290	28	0.95	40	1,500
1	<0.005	0.025	0.31	0.63	0.05	0.021	173
9	<0.005	<0.005	54	12	0.02	0.077	997
10	<0.005	0.94	40	7.6	0.10	2.3	1,630
11	<0.005	2.7	25	6.4	0.07	0.83	744
12	<0.005	0.13	2.5	0.99	0.04	0.17	274
21	0.006	0.77	39	7.4	0.12	4.3	591
36	<0.005	0.027	0.09	2.1	0.02	0.10	250
EF	0.01	1.0	0.3	None	0.7	0.02	None

planter mix, 1/3 manure, and 1/3 limestone (1, 10, 11, 12), it appears that a ratio of 10 g of substrate to 100 mL of mine drainage lacks sufficient organic material to effect vigorous sulfate reduction. Finally, it appeared that reaching the effluent limits for Cd, Cu, and Ni would be possible, but achieving the regulatory limits for Fe and Zn would be more problematic.

Treatment Decisions

The results of the laboratory study were encouraging enough that it was decided to forego bench-scale experiments and immediately design and construct a small pilot cell. However, there were still some questions to be answered on the selection of the substrate materials. In particular, the first set of local materials were not suitable for an anaerobic treatment system, and it was necessary to make another search for materials that gave good results in laboratory studies. In addition, the inocula of SRB came from Colorado sources and sources from northern California had to be located. Consequently, while the physical design of the pilot-scale reactor was being developed, another round of laboratory tests were performed to find suitable organic materials and sulfate-reducing inocula. Because the base substrate mix of manure, limestone, and planter's soil mix was effective in promoting the activity of SRB and removing contaminants, the search for local materials centered upon these materials. They were found, but had to be transported extensive distances.

The manure came from a dairy farm and appeared to be processed so that it contained primarily hay with very little soil. It tested positive as an inoculum for sulfate reducing bacteria; however, the growth of bacteria was slow. In the final designation of the substrate for the pilot reactor, it was decided to use the processed manure for the substrate formulation, but to include 10% of raw, unprocessed manure as the bacterial inoculum.

Pilot-Plant Study
Design of the Treatment Module

The passive treatment system module (PTSM) consisted of influent piping, substrate, and effluent piping installed in a welded steel tank. The rectangular tank was 10.9 m long, 2.7 m wide, and 2.7 m high and open at the top. The interior of the tank was covered with a bituminous polymer coating to protect the steel. The bottom of the tank was covered with a 15-cm layer of 1-cm diameter clean gravel. A network of 5-cm diameter perforated PVC pipe was constructed on the gravel layer. An additional 7 cm of gravel was placed on the exposed gravel and pipes. The top of the gravel was covered with needle-punched polyester geotextile fabric.

The substrate consisted of equal masses of processed manure, 1-cm diameter limestone, and dark fine-grained soil. The three components were blended and added to the PTSM in such a way that the substrate was not allowed to fall more than 1 m to minimize compaction. For inoculum, a portion of the substrate was blended with the raw manure and placed in the PTSM at the 1/3 and 2/3 level. A distribution network of perforated 5-cm-diameter PVC similar in configuration to the effluent piping was installed on top of the substrate. Water passes downward through the 2-m-thick substrate and is collected in the effluent piping located at the base of the tank. Flow through the system is controlled hydraulically by the elevation of the effluent pipe relative to the level of water within the module.

Table 4 Sulfide generation in mol/L for the passive treatment system module based on sulfate reduction. For comparison, the metals removal in mol/L is also shown.

Date	Sulfate (mg/L)		Sulfide (Molar)	Metals (Mn+Fe+Ni+Cu+Zn+Cd), (Molar)		
	In	Out	Produced	In	Out	Removed
9/24/91	2,900	1,100	0.0188	0.0083	0.0002	0.0081
10/8/91	2,600	2,600	0.0000	0.0091	0.0001	0.0090
10/21/91	3,000	2,200	0.0083	0.0083	0.0005	0.0079
11/5/91	2,700	2,300	0.0042	0.0087	0.0006	0.0081
12/3/91	2,600	2,200	0.0042	0.0087	0.0008	0.0070
1/8/92	2,600	2,100	0.0052	0.0084	0.0015	0.0069
2/25/92	2,700	2,100	0.0063	0.0090	0.0019	0.0071
3/24/92	3,200	2,600	0.0063	0.0137	0.0027	0.0109
4/28/92	3,000	2,300	0.0073	0.0109	0.0015	0.0093
5/26/92	3,000	2,200	0.0083	0.0106	0.0005	0.0101
6/30/92	2,900	2,400	0.0052	0.0102	0.0004	0.0098

Operation

The loading of the PTSM was based on the limiting reactant concept for loading of a sulfate-reducing reactor (Machemer et al. 1993, Wildeman et al. 1993). The premise is that the rate of generation of sulfide by the bacteria must meet or exceed the rate of flow of heavy metals (Mn + Fe + Cu + Zn) into the reactor. Based on previous studies (Reynolds et al. 1991, Wildeman, et al. 1993), the rate of sulfide generation was estimated to be 0.3 mol of S^{2-} per m^3 per day. The metals concentration in the influent totals ~ 800 mg/L. Therefore, using an average molecular weight of 55 g/mol for the metals in the drainage, this results in a metals influent concentration of 0.014 mol/L. Given the volume of the PTSM as 55 m^3, the total sulfide produced per day is 16.5 mol. Therefore, the flow was set so at 800 mL/min that the metals input would not exceed 16.5 moles/d.

To start the test, impoundment seepage was added until the substrate was thoroughly soaked and water breached the surface. Then, the system was left to incubate for 2 weeks. The sulfate in the seepage and the easily extracted organic compounds from the manure serve as excellent nutrients for the SRB. After 2 weeks, it was assumed that the system was an active bioreactor. The initial flow was set at 800 mL/min, and the PTSM was continuously operated for 22 months from October 1991 through July 1993. During this time the physical operation of the PTSM was free of problems.

For the first 10 month period, the analytical results on total constituents in the effluent are given in figure 1. In figure 2, the total concentrations of Fe in the influent and effluent as well as the dissolved Fe concentration in the effluent are shown. For the other constituents, except for excursions in Cu at the beginning of the study and Zn at the end of the period, the concentrations of dissolved constituents were only slightly less than the concentrations of total constituents. During this time, the composition of the influent water was essentially the same as that given in table 3, except in March when annual recharge of the aquifer increased concentrations of contaminants in the drainage by 10% to 70%. This increase can be seen for Fe in figure 2. In table 4, the analytical results of sulfate concentrations in the influent and effluent are presented. The molarity of heavy metals in the influent and effluent are also shown so that the balance between sulfide produced and metals removed can be compared.

At the mine site, Eh, pH, and temperature measurements were performed twice a week as part of the on-site maintenance program. The PTSM tank was built of steel and was above ground. In addition, the tank was not insulated . As a consequence, it responded quite well to changes in temperature as the seasons changed. Figure 3 gives the on-site values of Eh and temperature over the course of the 10 month study. Twice during the study, the temperature of the substrate at various depths and locations was determined. In both instances, the temperature range in the PTSM was less than 3° C with respect to both depth and lateral position.

Analysis of Operation

For the first 6 weeks of operation, removal results were excellent and followed the results achieved in the laboratory study. However, beginning in November, iron removal began to decrease in two respects. In the effluent,

a large difference between total and dissolved iron was observed, and the concentration of Fe increased in both the dissolved and total fractions. This increase could not be attributed to a change in the concentration of Fe in the mine drainage, which was approximately constant until March.

At least two properties of the PTSM operation can account for the change in dissolved Fe concentration. First, as has been shown in Colorado studies of sulfate-reducing reactors (Machemer and Wildeman 1992), in the first 6 weeks, two factors operate to promote metals removal. First, metals are readily removed by adsorption onto sites in the organic substrate. However, after 6 weeks, most of the sites are filled and this method of removal is no longer available; therefore, metal removal decreases. Also during the first 6 weeks, small molecular weight organic compounds are readily available to the SRB. This availability of nutrients causes large bacterial activities in the initial stages of operation. As seen in table 4, other than the anomalous value on October 8, the largest sulfate reduction values occur in the first 4 weeks of operation.

Startup in September, just as temperatures in the tank started falling, can also account for the increase in Fe concentration. SRB activity is known to decrease with temperature decreases, and Kuyucak et al. (1991) found the most drastic decrease when the temperature fell below 10° C. In the PTSM, the temperature fell below 10° C on about November 15 and did not rise above 10° C until about April 15. Fe concentrations would be most affected by a reduction in SRB activity because FeS is the form of iron sulfide formed and it is the most soluble of the acid-volatile sulfides (Machemer et al. 1993, Wildeman et al. 1993).

The reason for the increase in total Fe concentration is not known because material suspended in the effluent was not analyzed. If the increase was from geochemical conditions in the effluent, two reasons could best explain the increase. First, there was a high concentration of suspended organic material in the effluent, especially at the beginning of operation. Cu is strongly adsorbed onto organic material (Machemer and Wildeman 1992), and the large discrepancy between total and dissolved copper in the beginning of operation could be accounted for by suspended organic material. However, during the first month of operation, there is little difference between total and dissolved iron. Thus it appears that adsorption onto suspended organic material may not be the only reason for the increase in total Fe.

The other strong possibility for this difference is the presence of suspended ferric hydroxide in the water. Between pH 6 and 7, which are the operating pH's of the PTSM, ferric hydroxide would be stable at Eh's above 200 mV (Wildeman et al. 1993). The values of Eh in the effluent in figure 3 show that 200 mV was exceeded on about December 15. Consequently, this might account for the large difference between dissolved and total iron during the winter. However, when the Eh of the effluent falls below 200 mV on about May 1, there is still a difference between total and dissolved iron. This behavior suggests that the difference between total and dissolved iron is due to one or a combination of the following causes: (1) The difference is not caused by just one chemical constituent in the effluent water, (2) the difference may be caused by some physical condition in the PTSM that allows suspended Fe to remain in the effluent, or (3) the difference is caused by Fe precipitates still forming after the water has left the PTSM.
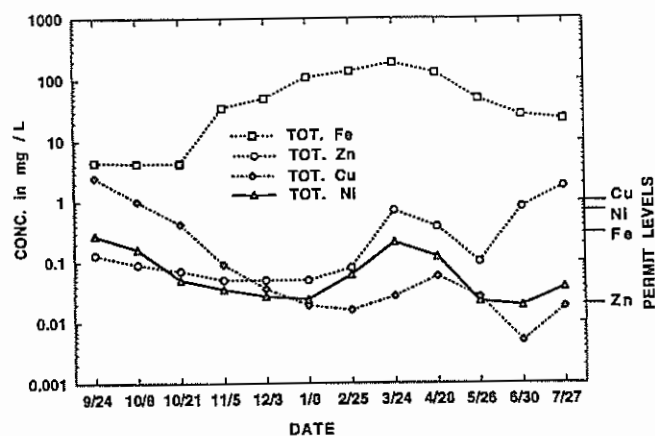

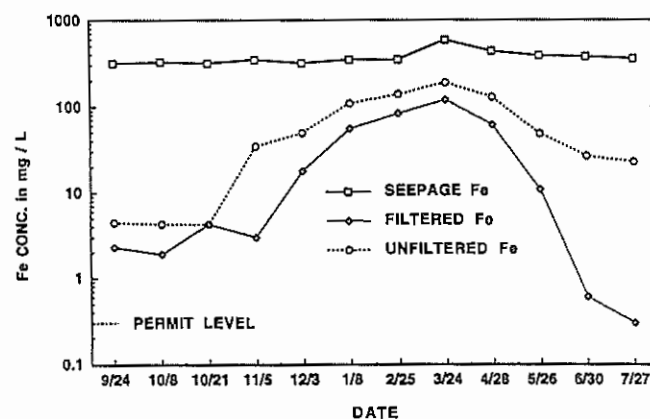Figure 1. Unfiltered effluent metal concentrations in mg/ L.

Figure 2. Influent (seepage) and filtered and unfiltered Fe concentrations in mg/L in the PTSM.
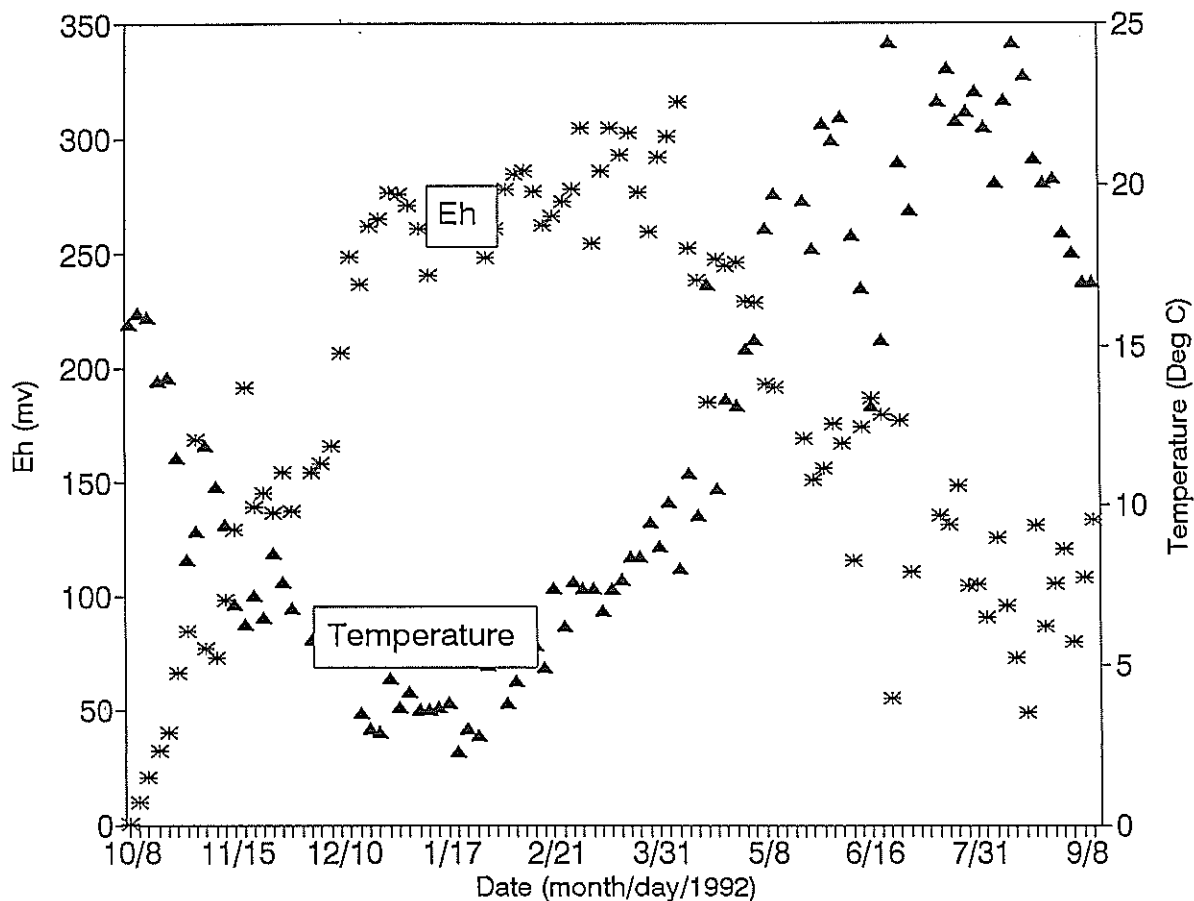
384

Figure 3. Variation of oxidation-reduction potential (Eh) with PTSM temperature in 1991 and 1992.

The frequent monitoring of the temperature and Eh along with the Fe and sulfate concentration values gives some insight into the activity changes of SRB with
temperature. For the PTSM, Eh increased as temperature decreased with little time lag during the fall. The highest Eh occurred in mid December at the lowest temperature. However, the Eh remained high until the end of March, whereas the temperature started rising at about the end of January. In table 4, examination of the sulfide produced implies that the decrease in the rate of sulfate reduction correlates well with decrease in temperature and increase in Eh. However, the peak in the Fe concentration was at about the time when the system is impacted by the increase in metals in the influent. During the time of reduced SRB activity, Ni, Cu, and Zn were consistently removed, quite likely because they form less soluble sulfides. Apparently, the capacity to remove metals continued even though sulfate reduction is diminished.

Conclusions

From this study, a number of conclusions can be made concerning the theoretical and practical development of anaerobic wetlands and reactors to treat mine drainage. The limiting reactant concept of balancing sulfide production with metal inflow appears to control the operation of the PTSM. In addition, when there is not enough sulfide generated, the concentration changes in the effluent can be predicted. Also, there appears to be some excess capacity in the reactor, so that if SRB activity is diminished or Eh increases, removal of Ni, Cu, and Zn will continue, but Fe removal may decrease. From the point of view of substrate development, other organic materials will work as well as mushroom compost as long as they contain a good source of SRB, contain a good supply of organic nutrients, and provide a near-neutral pH environment for bacterial growth. Finally, the expected relation between temperature, SRB activity, Eh, and contaminant removal has been established in the field.

On a practical basis, this study has shown that for anaerobic systems, a well-designed laboratory program will give an excellent indication of how effective removal will be. Also, laboratory experiments will provide the information needed to select the proper substrate materials and inocula. The information provided by these laboratory studies is adequate; thus for typical acid mine drainages, bench-scale studies are not necessary. From an operational viewpoint, nothing unusual occurred during the operation of the PTSM, and typical mine personnel were able to monitor and operate the system.

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