ARSENIC AND NICKEL REMOVAL BY WETLAND SEDIMENTS[1]

Andrew Fyson[2], Margarete Kalin[2], and Les W. Adrian[3]

Abstract: Laboratory experiments were carried out to test the capacity of sediments from muskeg ponds to treat waste rock seepages with mean concentrations of As and Ni of 36 mg/L and 79 mg/L, respectively (1992 data). Seepage water was added to column reactors containing muskeg sediment, and additions of organic matter (alfalfa, potato waste and hydroseeding mulch) were made to the sediments to stimulate microbial activity. In the first experiment, arsenic concentrations in the water column of the reactors decreased to less than 1 mg/L from 50 mg/L, and Ni to less than 0.1 mg/L from 74 mg/L in 112 days. A second experiment established that the results are reproducible. More than 90% of As and Ni present at start-up is removed within 43 days. A third experiment indicated that alfalfa and potato waste increased the rate at which reducing conditions were established, which in turn resulted in increasing the rate of arsenic and nickel removal from the seepage water. The addition of alfalfa resulted in greater metal removal than when potato waste was used. These experiments have shown that muskeg sediments have the capacity to remove As and Ni from a waste rock seepage water through providing conditions that facilitate precipitation and adsorption with or without the addition of organic amendments.

Additional Key Words: acid mine drainage, passive treatment, sulfate reduction, iron reduction.

Introduction

Waste rock piles from metal mining operations often generate seepages with low pH and high concentrations of heavy metals. Depending on decommissioning methods chosen, contaminated seepage waters may emerge from waste rock piles for a long time. Treatment may therefore be required in perpetuity. Passive treatment systems, utilizing natural processes driven by bacteria, are attractive treatment alternatives, as opposed to maintaining a chemical treatment plant. Such systems would provide a low maintenance option and are environmentally sustainable.

Research on the utilization of wetlands for the treatment of a variety of wastewaters has been carried out in the past decade, including constructed wetlands treating acid mine drainage (AMD) from coal operations (Brodie 1988, Hammer 1989). Wetlands are considered passive treatment systems, since they have the capacity to regenerate themselves through continued growth.

Microbially-driven sulfate and iron reduction are processes occurring naturally in wetland sediments which facilitate the removal of metals from the AMD through increasing the pH, which in turn results in precipitation of the metals either as hydroxides or as sulfides (Wildeman 1993, Kalin 1993). Adsorption processes, which assist in metal removal, can also be active in wetlands. Wieder (1992) has provided some estimates quantifying different metal removal mechanisms in wetlands. Eger and Lapakko (1989) utilized peat in constructed wetlands to treat AMD with Ni concentrations up to 15 mg/L, following laboratory studies where peat was observed to adsorb up to 20 g Ni/kg dry weight.

Kalin (1993) reported Ni concentration reductions in an AMD tailings seepage from an initial concentration of 25 mg/L originally to less than 1 mg/L. The microbially-driven treatment system removes Ni likely by co-

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[2] Andrew Fyson, Microbiologist and Margarete Kalin, Ecologist, Boojum Research Ltd., Toronto, Ont, Canada.

[3] Les W. Adrian, Environmental Engineer, CAMECO Corp., Saskatoon, Saskatchewan, Canada.

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precipitation with iron. When reducing conditions are established in the treatment system, Ni also potentially precipitates as a sulfide. Through additions of organic matter to the sediments, it should be possible to stimulate microbial activity and thereby maintain the capacity of the sediment to remove metals from water. If reducing conditions can be achieved in the sediment, a biological treatment system, for seepage waters which contact sediments in muskeg ponds, would be developed.

This paper presents the results of laboratory reactor experiments in which waste rock seepage water was added to sediments from muskeg ponds. The objective of this study was to determine the chemical and microbial processes which take place in the sediment and define the specific conditions necessary for metal removal.

Materials and Methods

Laboratory reactors were set up, constructed from acrylic columns with a diameter of 10 cm and a total volume of 2.5 L. The reactors were gas-tight and fitted with rubber stoppers as sampling ports. In the first set of experiments, sediment samples were supplemented with an amendment consisting of 10 % w/w of ground alfalfa and 90 % w/w of weathered hydroseeding mulch. Prior to addition to each column, 100 cm^3 of the amendment blend were mixed into 300 cm^3 of the sediment sample. The reactors were then filled with 1.0 to 1.5 L of test water, leaving approximately 0.5 litres of headspace in each reactor. The test water (station 6.11) is from a seepage collection pond next to the waste rock pile of a uranium mining operation in northern Saskatchewan, Canada. Three types of sediments from three different locations in two muskeg ponds in the same drainage basin of the waste rock pile were used in the first experiment. Water for the reactors was prepared by diluting waste rock pile seepage water with water to 20% of the original seepage concentration, since it was expected that, upon scale-up in the muskeg ponds, a dilution of the concentrations of most ions in the seepage would take place. To increase the arsenic concentrations in the seepage, the water was spiked with KH_2AsO_4 to a concentration of 50 mg/L arsenic to test the ability of the ecosystem to remove As at the maximum concentration likely to be encountered in the field.

A second experiment was set up using sediment from a single location. In this experiment, the same conditions were provided as in the first experiment, including the mixing of organic amendments into the sediment and using diluted (20%) seepage. This experiment was performed in order to establish whether the results obtained in the first experiment were reproducible.

During set-up of the third experiment, new samples of muskeg pond sediments without additions of organic matter (Control) and undiluted waste rock seepage water were added to the reactors. The sediments in the reactors were left for 6 days prior to addition of seepage water (760 mL), thereby allowing them to equilibrate. Those reactors receiving organic matter were treated with a surface layer of potato waste (5 g) or alfalfa (37.5 g). Application of a surface layer to the sediment surface was considered a realistic approach to simulating application of organic matter in field conditions.

To verify the function of sediments in the treatment process, 2 L glass jars with organic material but without sediment at the same ratio as used in the reactors were set up, using 760 mL of seepage water and 5 g potato waste or 37.5 g alfalfa. The quantities of organic matter added to the sediment were based on related work in base metal AMD (Kalin 1993).

Measurements of redox potential, electrical conductivity and pH were made using standard methods. The potential (Em), measured in the reactors, was converted to redox potential (Eh) by the following formula: Eh (mV) = Em (mV) + (241 - 0.66(T°C - 25)) to adjust for the potential of the reference electrode (Hem 1985). Water samples from above the sediment-water interface were extracted from the reactors through the sampling ports with a syringe. Samples were then filtered through 0.45 μm cellulose-acetate filters, acidified with concentrated HNO_3 and stored under ice or at 4°C until required for further analyses. Acidity and alkalinity were determined by titration with 0.01<u>N</u> NaOH (to pH 8.3) and 0.01<u>N</u> H_2SO_4 (to pH 4.5) respectively. Concentrations of Ni and As were determined by inductively coupled plasma spectrophotometry (ICAP) analysis and total Fe, PO_4, NH_4 and NO_3 were determined by colorimetric methods (Hach). Nickel was determined by the diacetyldioxamine colorimetric method (Merck). Arsenic was determined by the Merck test strip method. Sulfate was determined by the $BaSO_4$ turbidometric method (ASTM 4500-SO_4^{2-}).

Adenosine triphosphate (ATP) concentration, a measure of biomass, was determined with a firefly luciferase test. Cells were lysed with ethanol, filtered, further lysed with acetone, filtered and then ATP was extracted in trisodium phosphate buffer containing Mg-EDTA to prevent inhibition of the luciferase enzyme by heavy metal ions. Sulfate reducers were enumerated with Rapidchek II tests (Conoco, Houston, Texas) which detect the presence of adenosine phosphosulfate (APS) reductase by a colour-linked immunological assay.

Results

The water chemistry of the seepage used in the experiments for 1991 and 1992 is summarized in table 1. The average pH of this water was 4.28. This water is characterized by high concentrations of Ni and As. The seepage water also contains very high nutrient concentrations (ammonium, nitrate and phosphate). The sample used for the laboratory experiments had a Ni concentration of 74 mg/L, an As concentration of 50 mg/L and pH of 3.85.

Table 1. Major chemical parameters in waste rock seepage pumping pond water, 1992.

Parameter		Mean	Standard deviation	Minimum	Maximum	Number of samples
pH....................................	units	4.28	4.38	3.94	5.20	6
Conductivity.....................	uS/cm	1,433	569	259	2,120	6
Total dissolved solids.......	mg/L	774	524	210	1,360	4
Chloride............................	mg/L	3.0	1.8	0.8	6.0	6
Bicarbonate......................	mg/L	2.7	2.1	0.0	5.0	3
Sulfate..............................	mg/L	546	376	86	1,060	5
Calcium............................	mg/L	137	68	24	210	8
Iron..................................	mg/L	3.3	4.4	0.1	9.6	3
Potassium........................	mg/L	23	11.9	5.0	46	8
Magnesium.......................	mg/L	51	28	7	92	8
Manganese.......................	mg/L	3.9	1.8	0.9	6.5	6
Sodium.............................	mg/L	28	18.8	3.0	61	5
Ammonia-N......................	mg/L	4.6	2.4	0.8	7.2	6
Nitrate-N...........................	mg/L	87	39	12.0	128	6
Phosphate........................	mg/L	34	30	1.0	89	5
Total arsenic.....................	mg/L	36	32	5.4	97	5
Total nickel.......................	mg/L	80	74	4.8	220	5
Aluminum.........................	mg/L	14	27	0.1	68	5

The characteristics of the sediments are given in table 2. The sediments have a diverse microbial population. In two of the sediments, sulfate reducing bacteria could be quantified (1 to 50 x 10^4 cells/mL). Fine particulates plugged equipment which prevented quantification of sulfate reducing bacteria in the third sediment. The ATP values indicate the presence of active microbial populations in all 3 reactors at the time the reactors were set up.

The Ni and As concentrations after 87, 112 and 229 days in the first experiment are given in table 3 for each of the three sediment types used. Nickel concentrations declined in all three reactors to less than 0.12 mg/L by day 87. Thereafter, low Ni concentrations were maintained until the experiment was terminated after 237 days. Arsenic concentrations decreased substantially, in the presence of all three sediments, to 0.4 to 0.7 mg/L.

The results of the first experiment were encouraging. A second experiment was set up to determine the reproducibility of the results obtained. The data collected 115 days after set up are summarized in table 4. Eh values remained positive, but were sufficiently low that denitrification and dissimilatory nitrate reduction could proceed in the water column. These processes, which reduce nitrate to N_2O/N_2 and NH_4, respectively, can generate alkalinity and help establish conditions for further precipitation reactions, through lowering Eh and raising pH. The low NO_3 concentrations (0.41 to 1.94 mg/L) after 115 days also suggest that one or both of these processes has taken place. Sulfate reduction is indicated by the odour of H_2S and decline in the SO_4 concentration. As and Ni concentrations showed a rapid decline in all reactors. More than 90 % of both elements was removed within 44 days. After 115 days, As concentrations were in the range 0.1 to 1.3 mg/L and Ni concentrations were in the range of 0.05 to 0.14 mg/L. These values are in the same range as in the first experiment.

Table 2. Ecological and elemental characteristics of sediments.

Parameter		Sediment		
		BT1-200	BT2-250	BT2-350
Bacteria................	/mL	86,000,000	140,000,000	170,000,000
Sulfate reducers...	/mL	1000	50,000	ND[1]
Molds..................	/mL	410,000	410,000	1,800,000
Algae....................	/mL	1,500,000	1,400,000	1,900,000
ATP......................	ng/mL	54	51	33
Arsenic.................	ug/g	21	57	411
Nickel...................	ug/g	<97	195	786
Sulfur....................	ug/g	678	2,335	982
Iron.......................	ug/g	3,585	5,058	22,683
Phosphorus..........	ug/g	97	681	687
Aluminum.............	ug/g	10,756	13,132	102,121
Calcium................	ug/g	1,647	4,961	5,990
Magnesium...........	ug/g	1,550	2,043	11,881
Loss on ignition....	%	38	84	11

ND[1] Not determined

Table 3. Arsenic and nickel removal by sediments.

	As, mg/L			Ni, mg/L		
Sediment	87	116	229 days	87	110	237 days
BT2-250	3.00	1.10	0.40	0.12	0.11	0.05
BT2-350	1.90	0.20	0.40	0.06	0.10	0.03
BT1-200	0.60	0.75	0.70	0.06	0.06	<0.01

Table 4. Reactor water chemistry 115 days after setup.

Parameter		Reactor						
		E6-1	E6-2	E6-3	E6-4	E6-5	E6-6	Mean
pH at set up......	units	6.30	6.36	6.23	6.30	6.25	6.27	6.29
pH......................	units	6.44	6.55	6.73	6.07	7.00	7.00	6.63
Eh......................	mV	+88	+100	+163	+102	+129	+189	+129
Conductivity......	uS/cm	639	620	585	715	523	554	606
Iron....................	mg/L	5.86	5.15	1.38	7.30	1.20	1.56	3.74
Arsenic..............	mg/L	1.30	0.40	0.10	0.30	0.60	0.60	0.55
Nickel................	mg/L	0.05	0.05	0.08	0.08	0.06	0.14	0.07
Sulfate...............	mg/L	18	33	71	40	69	76	51
Acidity...............	mg/L	3.9	1.7	10.0	5.9	8.1	2.9	5.4
Alkalinity............	mg/L	190	223	231	205	192	208	208
Phosphate.........	mg/L	0.29	0.26	0.33	0.37	0.76	0.49	0.42
Ammonium.......	mg/L	1.5	0.7	2.2	6.5	1.4	1	2.2
Nitrate...............	mg/L	1.94	0.48	0.88	0.41	0.75	1.36	0.97

A third experiment examined the amount of metal removal when organic material was added as a surface layer over sediments, rather than mixing the organic material into the sediment.

Control Reactors

In the control reactors, where organic amendments were not added, only minor changes in the water chemistry were observed in 70 days. The pH slightly increased in the first 16 days and thereafter declined slowly to original levels of 4.2 (fig. 1a). Acidity (fig. 1b) and Eh (fig. 1c) remained fairly constant. The iron concentration initially decreased, then abruptly increased (up to 21 days) and decreased again (by 34 days) to near initial levels (fig. 1d). Nickel concentrations declined steadily during the course of the experiment (fig. 1e). Arsenic concentrations remained steady for 21 days and then declined (fig. 1f). The NH_4 concentrations remained steady (fig. 1h) whereas the NO_3 concentrations (fig. 1i) declined from 63 to 12 mg/L (34 days). The PO_4 concentrations decreased somewhat (fig. 1j).

Potato Waste Reactors

In the potato waste reactors, there was an increase in pH from 4.1 to 5.1 (mean of 2 reactors) from day 8 onwards to 34 days after set-up (fig. 1a). This trend continued and by day 64, the mean pH of the two reactors was 6. The Eh decreased during the first 16 days of the experiment (fig. 1c). The abrupt decline in Eh between 10 and 14 days after set-up occurred at the same time as the dramatic increase in acidity (fig. 1b) due to release of reduced iron, and possibly organic acids (from microbial decomposition of starch), from the sediment.

The dramatic increase in iron concentration observed between day 10 and day 21 (fig. 1d) was likely due to ferrous iron concentration increases diffusing from the sediment, following microbial iron reduction, and likely accounts for the reduction in Eh. A much smaller increase in iron (from 0.45 mg/L at day 1 to 3 mg/L at day 20) was observed in the control jars, indicating that some oxidized iron precipitates initially present in the solution were reduced, and/or that the potato waste contains minor amounts of iron.

The observed decreases in phosphate concentrations (fig. 1j) are likely related to the increasing pH over the course of the experiment. As pH increased, more HPO_4^{-2} dissociated into PO_4^{-3} and precipitated primarily with aluminum. Increases in NH_4 during the first 10 days (fig. 1h) can be attributed to the decomposition of protein in the potato waste. Decreases in NO_3 observed between 7 to 10 days (fig. 1i) were likely due to denitrification, facilitated by the decline in Eh and the provision of organic acids as electron donors and carbon sources. A reduction in nitrate concentration was also observed in the absence of a sediment (table 5).

Table 5. Chemistry of control jars without sediments.

Treatment	Days	pH	Eh, mV	Cond, uS/cm	Temp, C	Fe, mg/L	Ni, mg/L	As, mg/L[2]	SO4, mg/L	NH4, mg/L	NO3, mg/L	PO4, mg/L	Acidity, mg/L[3]	Alkalinity mg/L[3]
Potato waste	1	4.12	445	1,620	21.9	0.45	74	50	ND	16	60	59	136	0
	3	4.37	425	1,660	21.8	3.10	74	50	ND	21	51	45	111	0
	8	5.53	90	1,670	13.2	1.10	74	50	594	21	57	59	97	59
	20	4.28	306	1,590	21.6	3.10	64	80	540	12	.3	47	ND	0
	51	6.42	181	1,536	21.9	0.10	73	100	100	18	0	96	103	116
Alfalfa	1	4.44	444	1,510	21.9	0.31	74	50	ND	18	60	48	87	0
	3	5.94	-237	4,100	21.8	0.52	40	50	ND	36	51	57	487	ND
	8	5.72	71	6,120	13.2	3.30	61	50	342	36	7	62	649	2,161
	20	5.69	-119	5,990	21.8	0.40	53	95	160	69	<.2	42	ND	ND
	51	6.83	111	6,110	21.9	ND[1]	17	75	400	500	ND	47	291	2,155

ND[1]Not determined.

[2]Interference with sulfides suspected in ASTM 3500-As Mercuric Bromide Stain method.

[3]mg/L CaCO3 equivalent.

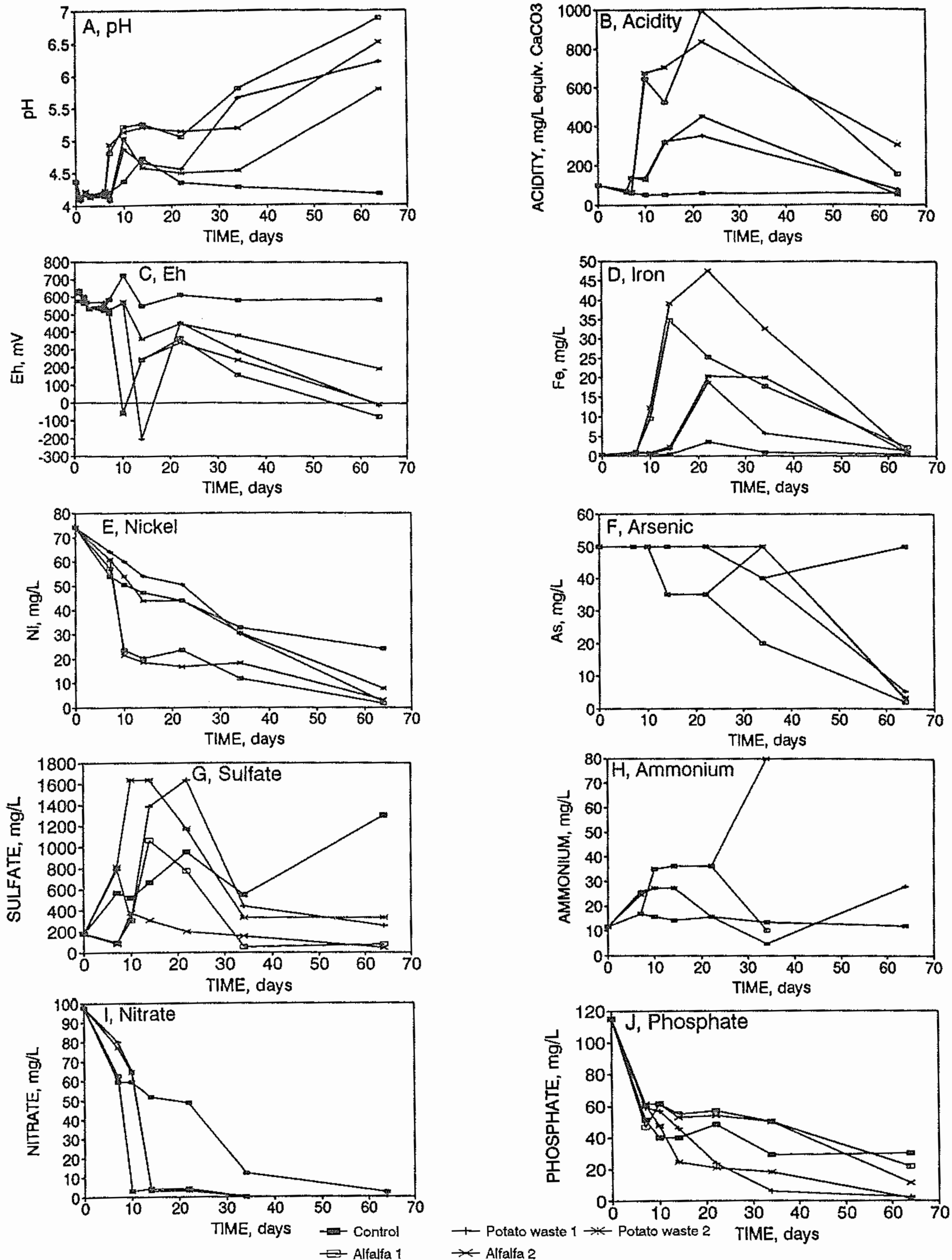


Figure 1. Changes in concentrations of major parameters over 64 days in third reactor experiment.

Ni concentrations decreased in the potato waste reactors over the course of the experiment at rates similar to those observed in the control reactor up to 30 days (fig. 1e). Arsenic concentrations held constant over the first 16 days (fig. 1f). From samples collected 63 days after set up, As concentrations had declined, averaging 4.1 mg/L. Sulfate concentrations increased in the reactors (fig. 1g). Sulfate was likely being released from both the potato waste and the sediments to the overlying water.

Alfalfa Reactors

Most trends of concentration changes in the reactors with added alfalfa were similar to those in the reactors with potato waste, except that concentration changes were more pronounced and changes occurred earlier in the experiment. The Eh decreased, within 2 to 3 days following set-up, to values much lower than in the potato waste reactors (fig. 1c). Greater NO_3 concentration decreases were observed (fig. 1i), more NH_4 was generated (fig. 1h) and higher pH's were reached (fig. 1a) than in the potato waste reactors. Phosphate concentrations declined more slowly than in the potato waste or control reactors, possibly due to release of phosphate from the decomposing alfalfa at a faster rate than precipitation (fig. 1j).

The measured iron concentrations increased (fig. 1d) and, according to the low Eh (fig. 1c), iron was likely in the reduced form, due to iron reduction by bacteria in the presence of alfalfa as a nutrient source. The nickel concentration decreases (fig. 1e) are likely related to production of large quantities of CO_2, with subsequent precipitation of Ni as $NiCO_3$. A consistent decrease in arsenic concentrations was not observed during the first 35 days of the experiment (fig. 1f). By 63 days however, the concentrations in the reactors had decreased to 2.49 mg/L As (mean of 2 reactors) with the exception of the control reactor. These decreases are likely related to the rate of microbial arsenate reduction and arsine production, which require low redox conditions for relatively long periods compared to precipitation reactions.

To determine the effects of the sediments in the metal removal process, and that of the organic material added to the sediment in the reactors, seepage water was tested in 1 L jars with additions of organic matter only. The results are summarized in table 5. After 51 days the organic matter had decomposed, the pH had increased and the concentrations of As, sulfate, nitrate, and phosphate, particularly in the jar with potato waste, had increased in the water. The possibility exists that interferences during colorimetric tests may account for the higher concentrations of As at this time.

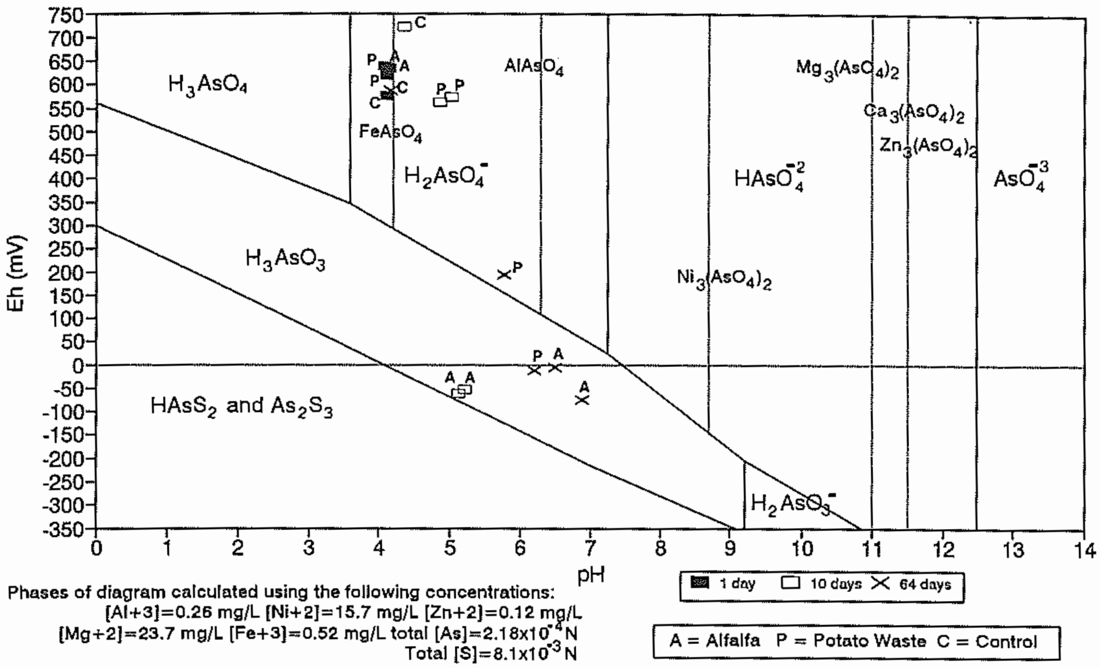
Discussion

Arsenic Removal Processes

Arsenic may exist in natural systems as many chemical species, owing to the fact that it two major oxidation states: +V and +III. The oxidation state of arsenic is dependent on the pH and the redox potential (Eh) of the system. A pH-Eh diagram was constructed to show possible forms of arsenic in particular chemical conditions (fig. 2).

At high Eh and low pH, arsenic +V exists as arsenic acid (H_3AsO_4). At higher pH, the As +V species, hydrogen arsenate ions ($H_2AsO_4^-$, $HAsO_4^{-2}$, AsO_4^{-3}) predominate in water. Also, at higher pH, these As +V ions are predominant over an increasingly wide range of Eh; at pH greater than 11, hydrogen arsenate ions are the only arsenic species in a system at Eh between -350 mV and +750 mV (Kotz and Purcell 1987, Stumm and Morgan 1981). Different metals can precipitate with arsenate ions above specific pH's. The average concentrations of metals and arsenic in three field enclosures are taken to calculate the pH of precipitation for these various compounds, including Fe, Al, Ni, Mg, Ca and Zn. They are shown as dotted vertical lines in the diagram.

The Eh values from the reactor experiments are plotted as they were changing over the period of the experiment, from the time at set-up (day 1), on day 10 and on day 64. On day 1, all the reactors lay within the



Phases of diagram calculated using the following concentrations:
[Al+3]=0.26 mg/L [Ni+2]=15.7 mg/L [Zn+2]=0.12 mg/L
[Mg+2]=23.7 mg/L [Fe+3]=0.52 mg/L total [As]=2.18×10^{-4} N
Total [S]=8.1×10^{-3} N

Figure 2. Redox-pH arsenic species phase diagram. Experiment 3 reactors' pH and redox on day 1, 10 and 64 are plotted.

zone where As occurs as $H_2AsO_4^-$. By day 10, the reactors which received organic material had changed considerably; the control and potato waste reactors were still in the $H_2AsO_4^-$ zone, whereas the alfalfa reactors were in the zone where the predominant form is H_3AsO_3. By day 64, one of the potato waste reactors had moved near to the AsO_3 zone, the zone where precipitation of As as sulfides is possible. The control reactor remained in the $H_2AsO_4^-$ zone for the entire 64 days.

Nickel Removal Processes

In aquatic ecosystems, the chemistry of nickel is less complex than arsenic, owing to its predominant oxidation state of +II. Therefore, there is both a more limited variety of chemical species of nickel, and fewer pathways by which dissolved nickel is removed from solution. Nickel is very mobile in acidic, high Eh water, while in high pH, reducing (low Eh) conditions, nickel sulfides can form. In addition, nickel is relatively amenable to adsorption onto iron and manganese hydroxides, clay particles and organic surfaces. Because of these properties, nickel removal can be anticipated in wetland environments with microbially active, reducing, organic sediments.

Summary and Conclusions

The reductions in arsenic and nickel concentrations observed in laboratory column experiments were also demonstrated in field enclosures installed in some of the same wetlands from which the reactor sediments were collected (Smith et al. 1993). Overall, field and laboratory estimates all fall within the same order of magnitude.

The laboratory experiments have established that muskeg sediments represent an effective micro-environment for removal of As and Ni from waste rock seepage water. The metal removal is associated with reducing conditions and is accelerated by the addition of readily degradable organic amendments which feed microbial processes.

The rate of arsenic removal was augmented by the presence of alfalfa, but not by potato waste. Both amendments reduced the Eh and augmented the supply of nutrients for anaerobic, alkalinity-generating microbial processes. This microbial community was active, as suggested by the decline in sulfate, the H_2S odour indicating sulfate reduction, the increases in dissolved iron (Fe III reduction), the dramatic decline in nitrate (denitrification) and the rise in pH. The removal of Ni in the reactors without amendments indicates that the processes involved were already present in the sediment. The addition of alfalfa increased the rates of As and Ni removal, compared to the other treatments. The rapid decline in Ni can be attributed to precipitation of $NiCO_3$ and/or NiS following the rise in pH generated by anaerobic microbial processes, including denitrification, iron reduction and sulfate reduction. Addition of alfalfa, both in the reactors and in the field, increased rates of Ni and As removal.

As a first approximation, the experimental results can be used to estimate the muskeg pond treatment capacity. A decrease of 49 mg in As concentration in the columns with a surface area of 0.005 m^2 in 65 to 110 days is equivalent to 151 $mg/m^2/day$ and 89 $mg/m^2/day$, respectively. For Ni, in all three reactors of the first experiment, nickel concentrations decreased from 12.9 mg/L to 0.2 mg/L in 40 days, equivalent to 64 $mg/m^2/day$. The rates for the third experiment range from 154 to 253 $mg/m^2/day$ for Ni and for As, 196 to 211 $mg/m^2/day$.

The data presented here, together with results from a field experiment (Smith et al. 1993), indicate that long-term exposure of seepage water to wetland sediments is a very promising approach to treatment of this wastewater. Ongoing experiments will determine the long-term stability of precipitates or other forms of the heavy metals held in the ecosystem.

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