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The amelioration of acid mine drainage by an *in situ* electrochemical method—I. Employing scrap iron as the sacrificial anode

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Abstract—The project began in an attempt to develop technology to ameliorate acid mine drainage (AMD) problems occurring at the Sherman Iron Ore Mine of Temagami, Ontario, Canada. A series of laboratory experiments were conducted to evaluate the effectiveness of an electrochemical approach to ameliorate AMD. An electrochemical cell was constructed using a block of massive sulphide–graphite rock from the mine site as the cathode, scrap iron as the sacrificial anode, and acidic leachate collected from the mine site as the electrolyte. The cell was effective at raising the pH of ≈ 41.0 L of leachate from 3.0 to a maintenance value of ≈ 5.6. This result was accompanied by a significant decrease in redox potential from > 650 to < 300 mV. Furthermore, iron sulphate precipitate formed, with a concomitant lowering of Al, Cd, Co, Cu and Ni solution concentrations. The study clearly demonstrated the proposed electrochemical approach to be a technically feasible and practical method of ameliorating AMD.

INTRODUCTION

Acid Mine Drainage (AMD) results from bacterially catalyzed oxidation of sulphide-rich mining wastes. The metal-rich acidic water poses a potential threat to aquatic ecosystems occurring within the vicinity of both operating and abandoned mining operations and is considered the single greatest environmental problem facing the Canadian mining industry.

In 1991, with the support of Dofasco Inc., we began research to develop technology to ameliorate acid mine drainage problems associated with the recently decommissioned (1990) Sherman open pit iron mine, Temagami, Ontario, Canada (Fig. 1). The deposit occurs within an Algoma-Type Banded-Iron-Formation (BIF). During mining, a band of sulphide-rich graphitic bedrock, described as sulphide-facies iron-formation, was exposed for approximately 2 km along the N side of the S Pit, one of 5 open pits occurring on the property. The sulphide-rich zone contains blebs, nodules, stringers, and massive lenses of pyrite up to 1.5 m wide and tens of metres long. The S Pit acidic waters were generated by the interaction of oxygenated surface water and groundwater with the sulphide-rich formation. The pH 3.0 waters contain environmentally unacceptable concentrations of Fe, Mn, Al and other metals (Table 1).

At its simplest, the oxidation of pyrite to produce AMD can be described by the following stoichiometric equation: (bacteria)

$$2FeS_2 + 7.5O_2 + 7H_2O \rightleftharpoons 2Fe(OH)_3 + 4H_2SO_4 \quad (1)$$

The FeS_2 (pyrite) weathers to form $Fe(OH)_3$ and H_2SO_4. Methods of dealing with AMD can be related to this reaction readily. One set of methods, including liming, organic/biological filtration systems (Dvorak *et al.*, 1991; Kuyuack *et al.*, 1991; Shelp *et al.*, 1994a) and wetlands (Kalin and Smith, 1991; Hammer, 1990; Karathanasis and Thompson, 1991) treats the products of the reaction. A more fundamental approach is to prevent the reactants on the left-hand side of the reaction from coming together. This approach includes bactericides (Sobeck, 1987), mineral coatings

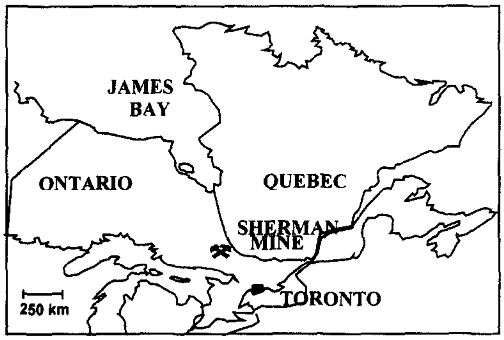

Fig. 1. Location of the Sherman Mine Field site.

Table 1. Chemistry of AMD South Pit, Sherman Mine (pH 3.0)

Conc, mg/L	Conc, mg/L
Fe...10	Ni...0.1
Al...10	SO_4^{2-}...1,500
Mn...5	Cl^{1-}...7
Zn...0.16	F^{1-}...1.4
Cu...0.1	

Source: Data courtesy of Sid Bartle, Dofasco Inc.

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on the surface of sulphide grains (Ahmed, 1991), engineered covers (Collin and Rasmuson, 1990; Broman *et al.*, 1991; Nicolson *et al.*, 1991), submergence (McCready, 1987; Ritcey, 1991), sphagnum bogs (Brown, 1991), iron-pan (Blenkinsopp, 1991; Ahmed, 1991), and biofilm (Blenkinsopp, 1991).

THE BASIS FOR AN ELECTROCHEMICAL APPROACH

The main objective of the current research was to evaluate an *in situ* electrochemical technology as an alternative treatment. Equation (1) represents a redox reaction. Referring to the associated reactions, electrons are produced by the oxidation of S^{2-} to SO_4^{2-} [eq. (2)] and the oxidation of Fe^{2+} to Fe^{3+} [eq. (3)]. The forward reaction rate can theoretically be slowed or even prevented, by supplying electrons through the incorporation of sulphide-rich rock as the cathode of an electrochemical cell and a material of greater electroactivity than pyrite as the sacrificial anode. The fundamental electrochemical reaction is a transfer of electrons from the anode to the cathode through the external circuit. Theoretically, this transfer should inhibit acid generating reactions both by creating a reducing environment on the surface of the sulphide mineral and, by reducing the activity of *Thiobacillus ferrooxidans* by increasing the pH of the microsites by the complexation of H^+ with O_2 to form water [eq. (5)], and/or through conversion of H^+ to H_2 [eq. (6)].

$$FeS_2 + 3.5O_2 + H_2O \rightleftharpoons Fe^{2+} + 2SO_4^{2-} + 2H^+ \quad (2)$$

$$Fe^{2+} + 1/4O_2 + H^+ \rightleftharpoons Fe^{3+} + 1/2H_2O \quad (3)$$

$$Fe^{3+} + 3H_2O \rightleftharpoons Fe(OH)_3 + 3H^+ \quad (4)$$

$$2H^+ + O_2 + 2e^- \rightleftharpoons H_2O \quad (5)$$

$$2H^+ + 2e^- \rightleftharpoons H_2 \quad (6)$$

A cell incorporating these principles, with pyrite as cathode and Fe as anode, would, in effect, pump electrons from anode to cathode and produce there a reducing environment. At the same time, reaction (5) [or under sufficiently reducing conditions reaction (6)] at the cathode, would raise pH. Conditions would be expected to drive towards the predominance field of ferric hydroxide (Fig. 2) which would then precipitate according to the coupled reactions (3) and (4). The continued addition of electrons from the anode would then be accommodated by the environment in the vicinity of the cathode following a path in Eh–pH space down towards lower redox conditions along a trajectory parallel to the precipitation boundary of $Fe(OH)_3$. If an alternative Fe^{3+} compound is precipitated, this will be indicated by a trajectory of different slope.

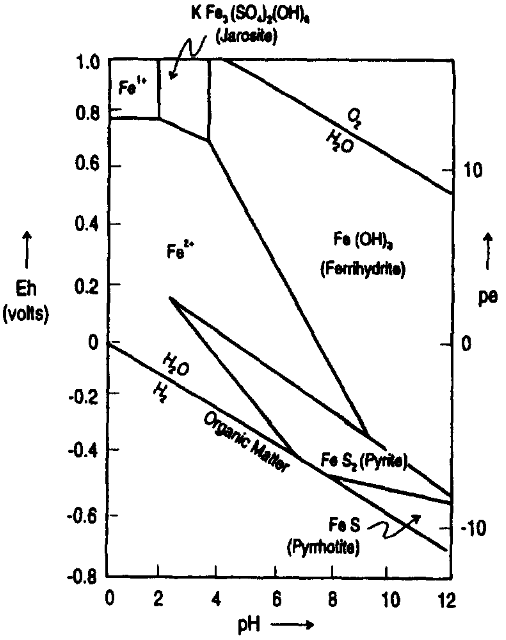

Fig. 2. Predominance fields in the system Fe–S–K–O–H for $a_{Fe} = 10^{-6}$, $a_S = 10^{-3}$, $a_K = 10^{-4}$.

EXPERIMENTAL WORK

A series of electrochemical cell experiments were conducted using a block of massive sulphide/graphite rock from the mine site as the cathode, scrap Fe as the sacrificial anode, and acidic leachate collected from the mine site as the electrolyte. Three experimental runs were completed using a scrap Fe sacrificial anode. Preliminary study results showed that the cell was effective at raising the pH of 41.0 litres of leachate from 3.0 to a maintenance value of 5.5 (Shelp *et al.*, 1994b). This result was accompanied by a significant decrease in redox potential from > 550 to < 300 mV. The formation of an iron sulphate hydrate precipitate, tentatively identified as coquimbite, $Fe_2(SO_4)_3 \cdot 9H_2O$, was accompanied by a concomitant lowering of Al, Ca and Mg solution concentrations. The chemical composition obtained by EDS analysis indicated an approximate atomic percent ratio of 43% S, 21% Ca, 18% Fe, 15% Mg, 1% K, 1% Mn, and 1% Si.

The experiment described in this manuscript was designed primarily to stop or significantly inhibit the acid generating reactions, both by creating a reducing environment on the surface of the sulphide minerals and by increasing the pH of AMD by converting H^+ to H_2. The effects of the rise in pH on possible coprecipitation of metal ions is examined.

Figure 3 is a schematic diagram of the experimental cell used in the studies. The sulphide/graphite block (45 cm by 30 cm by 30 cm) containing between 10–15% pyrite was of fissile character with minor fractures extending through the thickness of the block. A 3 cm head of water was maintained in the upstream pond to ensure that acidic leachate generated during the experiment collected in the downstream pond. The upstream and downstream ponds contained 27.5 and 41.3 L, respectively.

The sacrificial anode, suspended in the downstream pond, was attached to the semi-conductive sulphide–graphite block, the cathode, by a Cu plug assembly (Fig. 3). The downstream pond was purged with N_2 to maintain the dissolved O_2 content between 1.0 and 1.2 mg/L thereby inhibiting the formation of oxide coatings on the surface of the anode.

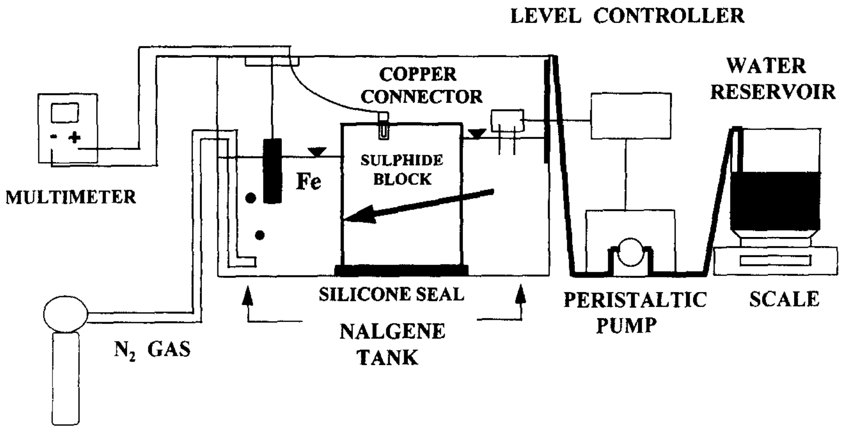

Fig. 3. Electrochemical cell setup used to test the feasibility of ameliorating AMD by cathode protection. The arrow shows the direction of flow from the upstream pond to the downstream pond. (Chemical analyses were conducted on the leachate contained within the downstream compartment.)

Scrap Fe was selected as the most suitable anode for 2 reasons. It is environmentally safe under natural conditions and it is relatively inexpensive. For Fe or any other anodic material to function effectively, the cell must be capable of raising and maintaining the leachate pH above 5.5, the theoretical pH of rain water.

A multimeter was used to record EMF and current on a daily to weekly basis throughout the experiment. The measured potential (Eh) and pH were measured using an Accumet 1000 Series meter with a Ag/AgCl reference electrode. The measured potentials were standardized to the hydrogen scale.

Water samples were taken periodically and analyzed for total dissolved metals such as Al, Ca, Cd, Co, Cr, Fe, K, Mg, Mn, Na, Ni, Pb, and Zn, by atomic absorption spectroscopy (AA) and inductively coupled plasma spectroscopy (ICP-AES), and anions by ion chromatography. Precipitate samples were collected periodically for X-ray diffraction, scanning electron microscopic and elemental analysis. X-ray diffraction analysis was conducted using a RIGAKU D/Max-A series powder X-ray diffraction system with specimen being step-scanned from 3° to 90°2θ, in 0.02°2θ increments with 4 s integration times. Precipitate samples were coated with 20 nm Au and examined with a Cambridge 250 scanning electron microscope equipped with a Noran thin window energy dispersive detector and a Tracor 5500 analyzer (SEM-EDS). Precipitate sub-samples were analyzed for total metal content by ICP-AES following dissolution with aqua regia.

RESULTS AND DISCUSSION

AMD is a problem in the environment not only because of acidity, but also because the solution contains metal species at concentrations mimical to living things. The rise in the pH of leachate treated by the proposed electrochemical cell process is directly relatable to the formation of precipitates such as Al and Fe hydroxide phases. The formation of precipitates should be accompanied by a diminution of toxic species through coprecipitation. These phenomena can be demonstrated by monitoring EMF, current, pH, Eh, by measuring the content of metals, such as Al, Cd, Co, Cu, Fe, Ni and Zn, and anions in the aqueous phase, and by collecting and analyzing the precipitate.

EMF and current

The cell generated a starting EMF measuring 870 mV. The EMF trend over time has 2 distinct segments: a relatively steep slope averaging −32.5 mV per day for the first 6 days and shallower slope averaging −10.0 mV per day for the next 38 days (Fig. 4). Starting at day 45, a steady state value of approximately 292 mV was recorded (data not shown). Segments 1 and 2 can be expressed by the following regression equations: EMF = −33.2143T + 902.79 and EMF = −8.93T + 738.89, respectively. The current decreased from an initial value of 4.9 mA to level off at 2.3 mA by day 12 (Fig. 5). This decrease was followed by a resurgence to 3.5 mA by day 37. These results may be explained by the analogy of a battery discharging as the electrolytic properties of the solution decrease.

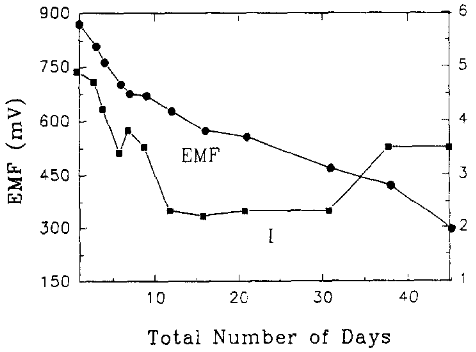

Fig. 4. Changes in EMF and current with time for the electrochemical cell experiment utilizing an Fe anode.

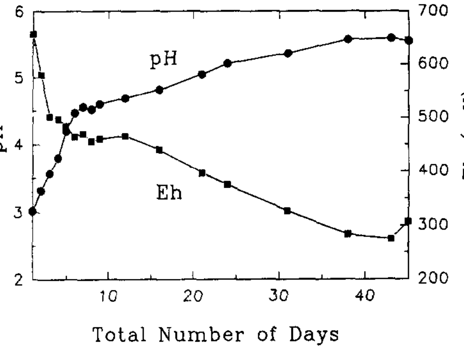

Fig. 5. Changes in Eh and pH with time for the electrochemical cell experiment utilizing an Fe anode.

pH and redox

The pH increased from 3.0 to 5.6 within 35 days. A bimodal reaction rate for the reduction of H^+ to H_2 is shown on Fig. 5. During the first 5 days, an average of approximately 1.8422E-4 mol H^+/L were reduced daily resulting in a pH of 4.47. During the following 30 days, the average daily rate was approximately 1.0398E-6 mol H^+/L per day. A possible explanation for the apparent bimodal reaction rate is that at low pH electrons are, for the most part, consumed by the residual H^+ ions of the system. As the pH rises, however, additional H^+ is generated through the hydrolysis of metal ions, specifically Al and Fe hydrolysis reactions [eqs (7) and (8)] are examples of heterogeneous hydrolysis reactions [Stumm and Morgan, 1981)].

$$Al^{3+} + H_2O \rightleftharpoons Al(OH)_3 + 3H^+ \tag{7}$$

$$Fe^{3+} + H_2O \rightleftharpoons Fe(OH)_3 + 3H^+ \tag{8}$$

Consequently, the rate at which the H^+ ion is converted to H_2 decreases until a constant pH is achieved. The reaction kinetics are further complicated by the reduction in the current over time as a result of the decreasing electrolytic properties of the leachate owing to the consumption of H_2SO_4.

The Eh of the system decreased from an initial value exceeding 657 mV to approximately 275 mV (Fig. 6) producing Eh/pH conditions which should inhibit the oxidation of sulphide minerals. The decrease in the measured redox potential, −123 mV/pH unit, exceeded the theoretical slope, −59 mV/pH unit, for the precipitation of $Fe(OH)_3$ derived from the Nernst equation, by a significant amount (Fig. 6). This suggests that $Fe(OH)_3$ may not be the controlling phase, that is that a precipitate of a different stoichiometry might be determining the path of the solution. Other factors such as a changing ionic strength of the solution during the experiment or the presence of reduced ion species would also affect the slope.

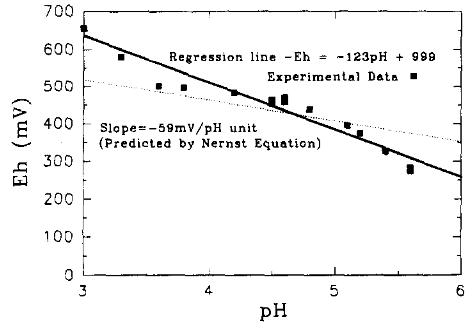

Fig. 6. Eh/pH diagram comparing experimental data with values predicted for the precipitation of $Fe(OH)_3$ using the Nernst equation.

Solid phase

At pH 5.3, a reddish-brown colored secondary solid phase was observed precipitating on the bottom of the experimental tank. Upon drying, 2 distinct mineral phases could be identified. A white mineral, measuring up to 0.5 cm in diameter and composed of radiating elongated crystals was observed superimposed on a reddish-brown amorphous groundmass [Fig. 7(a)]. Scanning electron microscopic examination clearly shows the radiating nature of the elongated crystals and the poorly crystalline to amorphous spherulitic nature of the groundmass [Fig. 7(b)–(d)].

Three predominant d-spacings at approximately 0.427, 0.758, and 0.306 nm, characteristic of gypsum, $CaSO_4 \cdot 2H_2O$ (Table 2) were obtained by X-ray diffraction. The chemical composition of the crystals obtained by EDS analysis indicated an approximate atomic percent ratio of 52.5% Ca and 47.5% S (Fig. 8). The approximate atomic percent ratio of the ground mass was 35.4% S, 28.6% Fe, 24.4% Mg, 7% Al, 2.6% Si, 1.5% Mn, and 1% K (Fig. 9). The quantitative data are presented on a non-oxide basis. Assuming that S is present as SO_4 the obvious electrical imbalance indicates that a multi-phase system exists with oxide or oxyhydride minerals constituting a significant portion of the groundmass. The contents of the trace elements Co, Cu and Zn measured 60, 183, and 806 mg/kg, respectively. Cadmium and Ni occurred in concentrations of less than 2.5 mg/kg.

Table 2. List of d-spacings and corresponding relative intensities for the precipitate groundmass

d-spacings (nm)	Relative intensities
0.758	83
0.427	100
0.380	36
0.306	69
0.287	24
0.268	21

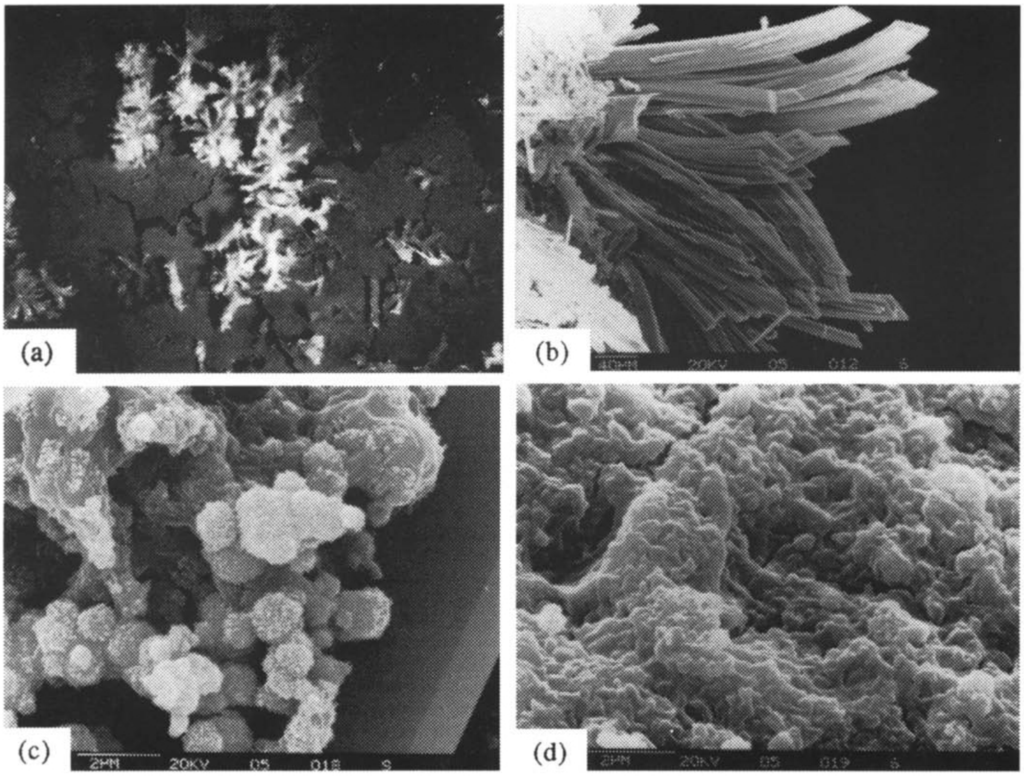

Fig. 7. Photomicrographs illustrating (a) crystals superimposed on an amorphous groundmass and scanning electron micrographs illustrating (b) radiating elongated crystals, (c) spherules occurring at the base of the crystals, and (d) the amorphous nature of the groundmass.

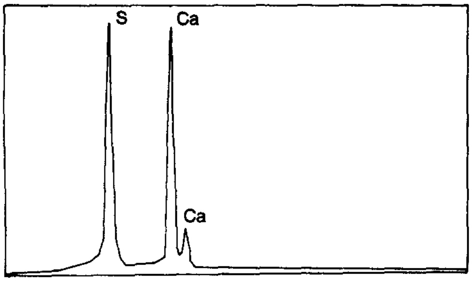

Fig. 8. Energy dispersive spectrum of the crystal phase for the electrochemical cell experiment utilizing a scrap Fe anode.

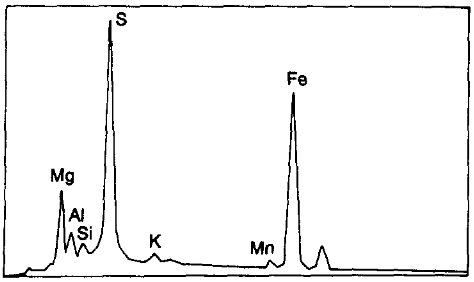

Fig. 9. Energy dispersive spectrum of the amorphous groundmass for the electrochemical cell experiment utilizing a scrap Fe anode.

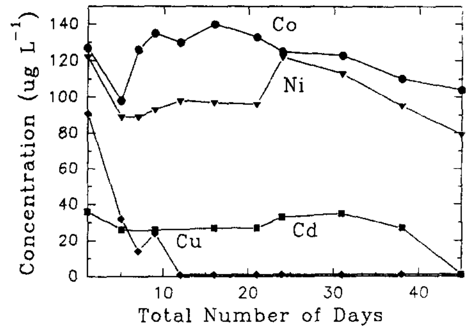

Fig. 11. Changes in Cd, Co, Cu and Ni with time for the electrochemical cell experiment utilizing a scrap Fe anode.

Chemistry of the solution phase

The general solution concentration trends for Al, Fe, and Mn; and Cd, Co, Cu, and Ni, with time are shown in Figs 10 and 11, respectively. All elements, excluding Fe, show significant decreases with time, for example Al, Cu, and Cd, or remain relatively constant, for example Mn. The Fe concentration increased to a steady state value of approximately 70 mg/L after day 25 (Fig. 10). The concentrations of SO_4^{2-}, Cl^-, and F^- remained relatively constant at 1400, 10, 2 mg/L, respectively, with time (data not shown). Expressing the elemental concentration data with respect to changes in solution pH, the concentrations of Al, Cd, and Cu showed the most significant decreases with increasing pH (Fig. 12). Copper decreased linearly from an initial concentration of 90 ppb to < 1 ppb by pH 5.6 (Fig. 12). In contrast, Al and Cd remained relatively constant at pH <4.5. At pH 4.5, Al showed a dramatic decrease from 16 ppm to < 1 ppm. Cadmium showed a similar trend at pH 5.5 with the concentration decreasing from 40 ppb to < 1 ppb.

The decrease in Al may be owing to either the precipitation of a separate Al phase, or by coprecipitation with Fe, through absorption of Al by, or incorporation of Al, into the Fe rich precipitate. The results suggest that Al may have precipitated as a separate phase (Fig. 12). Similarly, Fe may have precipitated as a separate phase as suggested by the 43% Fe contained in the groundmass. The expected reduction of Fe in solution with an increase in pH, however, was masked by the rising Fe concentration resulting from the dissolution of the scrap Fe anode (Fig. 12).

The concentration of Cu in solution may be controlled by coprecipitation adsorption processes associated with an Al precipitate or by the plating of Cu on the anode. Cadmium, however, is likely coprecipitating with Fe as an adsorbed phase. The order in which these metals precipitated appears to agree with the published scientific literature. In recent years, several review papers have been published describing the chemical reactions controlling heavy metal solubility in soils and aqueous systems, and the

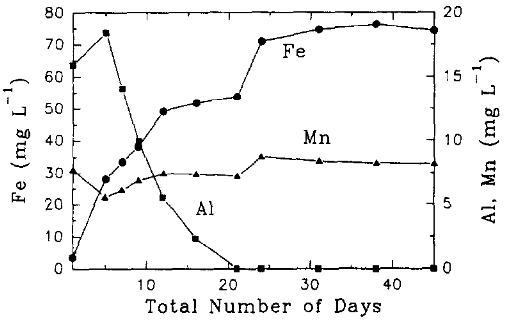

Fig. 10. Changes in Al, Fe, and Mn with time for the electrochemical cell experiment utilizing a scrap Fe anode.

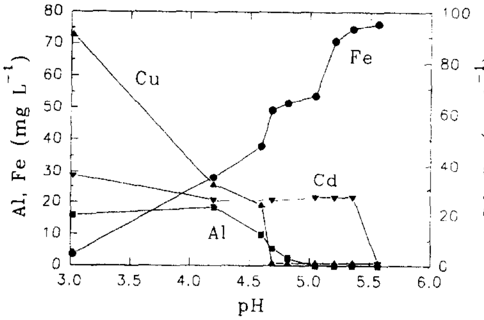

Fig. 12. Changes in Al, Cd, Cu and Fe with pH for the electrochemical cell experiment utilizing a scrap Fe anode.

associated environmental implications (Davies, 1980; Evans, 1989; MacBride, 1989, 1991; McLean and Bledsoe, 1992). Considerable discussion has focused on the adsorption of metals onto the surfaces of secondary mineral phases, such as oxides, hydroxides, and oxyhydroxides, of Fe and to a lesser degree Al. The degree to which a metal is adsorbed is dependent on many factors including metal speciation, and the intrinsic and conditional properties of the surface complexation site. Relative orders of metal affinity for adsorption sites have been determined for the various precipitate phases. For example, the relative order of sorption of some divalent metals for amorphous Fe hydroxides is Pb > Cu > Zn > Ni > Co > Cd; whereas the order is slightly different for $Al(OH)_3$: Cu > Pb > Zn > Ni > Co > Cd (Kinniburgh *et al.*, 1976). Similar metal affinity relationships have been predicted from the first hydrolysis constant of the metals as discussed by MacBride (1989).

CONCLUSIONS

The results of this laboratory study clearly demonstrate that the proposed electrochemical concept is an effective treatment for acid mine drainage. The electrochemical system has been shown to be capable of generating sufficient voltage and current to raise and maintain the pH of acid leachate at an environmentally acceptable level of 5.6 and to significantly reduce the redox potential, thus inhibiting the oxidation of sulphide minerals. Furthermore, as an added advantage, potentially toxic elements such as Al, Cd, Co, Cu, Ni, together with other trace elements, may be scavenged from solution by the formation of a relatively stable Fe/Al sulphate precipitate.

Future research will focus on an examination of dissolved O_2 on leachate chemistry and the nature of solid precipitates and their affinity for contaminant metals. Other possible anodic materials will also be examined.

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