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PREDICTION OF ACID MINE DRAINAGE POTENTIAL OF GEOTHERMAL SOLID WASTES

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ABSTRACT

Geothermal solid wastes such as scale and sludge from the Philippines and Mexico containing Cu, Zn, and Pb at levels above earth's crustal abundance were studied for their acidification potential if disposed in a landfill. A batch reactor technique using the iron and sulfur oxidizing bacteria *Thiobacillus ferrooxidans* was developed for geothermal wastes to predict their acid mine drainage and bioleaching potential. It was observed that almost 100% of Cu and Zn in the Mexican scale and less than 2% in the Philippine scale and sludge were released while Pb, the regulated element, was not found in the leachate which probably precipitated as PbSO,. This indicates that these geothermal residues likely will not be a threat to the environment due to their silicate nature and can safely be disposed in a landfill.

INTRODUCTION

In mineral mining, the prediction of acid mine drainage (AMD) is needed to find out if the quality of waters draining from a mine site will exceed environmental regulatory standards, and if so, what mitigation measures have to be provided at the outset. Accurate prediction of AMD is required both to protect the environment and to ensure that resources are expended wisely to prevent or control AMD. The experience in the mining industry on prediction of acidification potential and metal release will be useful also to the geothermal industry where solid residues with components (silica and metal sulfides) similar with mine tailings are produced (Peralta et al, 1996a). Earlier works identified the Philippine scale and sludge and the Mexican scale as having preliminary acid mine drainage potential using a static test called BC Research Initial Test (Peralta et al, 1996b). This required confirmation using a kinetic test involving microbial reactions.

The purpose of this study is to confirm the acid mine drainage potential of several geothermal residues and their amenability to landfill disposal.

Acid Mine Drainage

Acid mine drainage (AMD) is a problem commonly found in coal and metal mines whereby sulfide materials rejected during the mining of coal or minerals and deposited in mine tailings or heaps are oxidized to sulfates. This in turn releases runoff with high acidity and heavy metals causing pollution to the environment over a long period of time (Ferguson and Erickson, 1988; Atlas and Parks, 1993; David and Nicholson, 1995). Acidic drainage also causes severe corrosion problems to mining and ancillary equipment (CANMET, 1991). In western USA, the Forest Service estimated that between 20,000 and 50,000 sites (including abandoned and operating mines) are currently generating acid on forest lands and that drainage from these mines is affecting between 8,000 and 16, 000 km of streams (USEPA, 1994). The annual volume of acid-generating waste rock or tailings produced by the Canadian mineral industry is estimated at 140,000 dry tonnes/year (MacDonald et al, 1989). The sulfide oxidation is microbially enhanced by the presence of iron and sulfur oxidizing bacteria such as *Thiobacillus ferrooxidans*, that can survive at low pH (< 3.5) and high temperature (up to 60 ^{o}C). Since geothermal residues possess characteristics resembling mine or rock tailings and have also been traditionally disposed in open dumps (Peralta et al, 1996a), the investigation of possible AMD potential was most prudent.

Iron Oxidizing Bacteria

The reaction rate causing AMD is greatly accelerated by the presence of *T. ferrooxiduns,* to as much as 10^6 - fold (Singer and Stumm, 1970; Stumm and Morgan, 1981). These bacteria promote indirect oxidation of pyrite and other sulfides through the catalysis of the oxidation of ferrous ion to ferric ion which is an effective oxidant. However, they may also catalyze direct oxidation of sulfides by oxygen. These organisms act only as redox catalysts; they do not oxidize substrates or reduce oxygen but mediate the reaction or electron transfer. In doing so, they obtain a source of energy from these energy-yielding redox reactions for their metabolic needs. *T. ferrooxiduns* tends to live in environments such as hot springs, volcanic fissures, and sulfide deposits as well as oil brines, coal water, mine wastes, peat soil, concrete and building stone (Brock, 1970; Rossi, 1990).

Microbial Leaching

Due to biochemical reactions, insoluble metal sulfides can be degraded to soluble metal sulfates by direct and indirect methods of bacterial metabolism **(Stumm** and Morgan, 1981; Bosecker, 1984; Tyagi and Couillard, 1989). In the direct mechanism, the metal sulfide is oxidized to metal sulfate :

$$MeS + 2\,O_2 \xrightarrow{\text{bacteria}} MeSO_4 \qquad (1)$$

where Me is a bivalent metal. The heavy metal sulfides such as NiS, ZnS, CoS, PbS, and CuS are generally insoluble in aqueous acid leach media, while their sulfates have solubility with the exception for lead sulfate which is sparingly soluble (K_{sp} of 1.6×10^{-8}).

In the indirect mode of bacterial action, the metal sulfide is oxidized by a ferric ion without the direct participation of bacteria as shown below in Equation 2. This reaction takes place geochemically under conditions of weathering and leaching. Ferrous ions can be reoxidized as in Equation 4 and again ferric ions can act as the oxidizing agent. In indirect leaching processes, *T. ferrooxiduns* catalyses ferrous ion oxidation which takes place very slowly under normal conditions (Equation 4).

$$MeS + Fe_2(SO_4)_3 \longrightarrow MeSO_4 + 2\,FeSO_4 + S^0 \qquad (2)$$

The elemental sulfur that has been set free in Equation 2 will be oxidized to sulfuric acid mediated by bacteria in the following manner:

$$S^0 + 1.5\,O_2 + H_2O \xrightarrow{\text{bacteria}} H_2SO_4 \qquad (3)$$

In the same manner, the ferrous ion is reoxidized, mediated by the microorganisms to ferric ion:

$$2\,Fe^{2+} + \tfrac{1}{2}\,O_2 + 2\,H^+ \xrightarrow{\text{bacteria}} 2\,Fe^{3+} + H_2O \qquad (4)$$

and the iron redox cycle is repeated. The production of sulfuric acid will decrease the pH, which will enhance **further** the solubilization **of** metals. It is also possible to form a yellow insoluble precipitate called jarosite, $KFe_3(SO_4)_2(OH)_6$ which can hamper transport phenomena by coating the mineral. In bacterial leaching systems, it is desirable to prevent jarosite generation because of the formation of diffusion barriers on mineral surfaces and the scavenging of metal ions from the leach solution (Tuovinen, 1990).

AMD Confirmation Test

A test widely used in Canada and the **USA** is the B.C. Research Confirmation Test which requires inoculation with ***T. ferrooxidans*** to stimulate the rapid stage of oxidation (Bruynesteyn and Hackl, 1984; Lawrence et al, 1989). The sample (10-20 g depending on sulfur content) is placed in 250 mL Erlenmeyer flask with **70** mL nutrient media, 5-10 mL culture of *T. ferrooxiduns* at pH *2.2* - 2.5. The flask is placed on gyratory shaker at 35 °C in a CO_2 -enriched atmosphere, pH is monitored and additional sample provided. If the pH rises substantially, then the sample is considered nonacid producer. If the pH is <3.5, then sample is a potential acid producer. The basic limitations of this procedure are : (a) there is no specified procedure to spawn an acclimatized bacteria culture, (b) there is no assurance **of** bacterial growth as $FeSO_4$ was withheld from the culture media and bacterial viability was only checked at the onset and not periodically during the test, and *(c)* redox potential (Eh) and metal concentration of solution are not measured to indicate chemical reaction and metal release. This test was developed originally for mine tailings where the particle size is small, with high porosity, and containing larger amounts of sulfides accessible for bacterial attack.

To confm the acid mine drainage potential if disposed in a landfill, a series of experiments was carried out to determine (a) the best growth environment and medium for the *T. ferrooxidans,* (b) the appropriate procedure for the geothermal wastes, and (c) kinetic information on the acidification of geothermal residues. The B.C. Confirmation Test

(CANMET, 1991) which was found deficient in its nethod and monitoring scheme as discussed above, was modified and an acid mine drainage potential (AMDP) test was developed for the geothermal residues. The effect of agitation, temperature, and sterilization on metal leaching and bacterial growth was investigated using this AMDP test.

EXPERIMENTAL

All chemicals, salts, acids, and buffers used were of analytical grade while all solutions, standards, and dilutions were prepared using deionized water.

Bacteria Culture and Medium

The growth medium for the *T.* ***ferrooxidans*** (ATCC 19859) was modified from the standard laboratory technique of the American Public Health Association (APHA, 1992). This was popularly known as the 9K medium (Silverman and Lundgren, 1959) which was adopted by the APHA. The two modifications made in this work were the reduction of the FeSO, content to half the original formula and use of membrane filtration (0.45 μm pore size cellulose acetate) for sterilization of solution instead of autoclaving. The detailed procedure is described elsewhere (Peralta, 1997). The modified medium had the following constituents as shown in Table 1 below.

Table 1 Culture Medium for *T.* ***ferrooxidans***

Basal salts: in a 1 L Erlenmeyer flask	Amount
Ammonium sulfate $(NH_4)_2SO_4$	3.0 g
Potassium chloride KCl	0.10 g
Dipotassium hydrogen phosphate K_2HPO_4	0.50 g
Magnesium sulfate $MgSO_4.7H_2O$	0.50 g
Calcium nitrate $Ca(NO_3)_2$	0.01 g
Sulfuric acid, 10 N H_2SO_4	1.0 mL
Distilled water	700 mL
Energy source: in a 500 mL Erlenmeyer flask	
Ferrous sulfate $FeSO_4.7H_2O$	22.11 g
Distilled water	300 mL

Acclimation **of** Inoculum

A critical stage of the acid mine drainage potential test (AMDP) is the acclimatization of the pure bacteria culture to the specific samples to be tested. To prepare a viable culture as inoculum and which will survive throughout the duration of the test, a series of acclimation steps was designed at room temperature (23-25 ^{0}C) and without agitation. For each culture, ***5*** mL inoculum was used per 100 mL of the medium. At the onset, a medium shown in Table 1 but using 44.22 g/L of ferrous sulfate (APHA, 1992) was used on the pure culture (B_0) inoculum ***of*** *T.* ***ferrooxidans*** (ATCC 19859). Afterwards, the resulting culture (B,) was used as inoculum to a 100 mL fresh medium with the addition of 2 g ground sample (120 mesh) to be tested to obtain an acclimatized culture (B_2). Finally, **B_2** was used on a freshly made culture media containing 22.11 g/L ferrous sulfate and 2 g sample to produce B_3 culture that is ready as inoculum for the AMDP test. Prior to this, several experiments were performed to obtain the best conditions for high bacterial density and motility. These experiments were carried out with and without agitation, room temperature (23 ^{0}C) and inside incubator (35 ^{0}C), various amounts of ferrous sulfate in medium as well as several pulp densities (weight of sample over volume of solution). The success of each experiment was determined qualitatively through bacterial viability (density and motility).

Acid Mine Drainage Potential Test

After literature review and preliminary testing of the BC Research Confirmation Test, the following acid mine drainage potential (AMDP) procedure was developed to study the amenability of geothermal residues to land disposal. A brief description of the procedure follows. The *dry* samples were pulverized in a mortar and pestle to pass a 120 mesh Tyler screen and stored in air tight bottles prior to use. In triplicate, 100 mL of culture medium was poured slowly to 2 g of ground sample in a labelled 250 mL Erlenmeyer flask. The flask was plugged with nonadsorbent cotton wrapped with gauze. The flask was swirled manually and the pH was checked. If the pH was above 2.8, a few drops of 10N H_2SO_4 were added until stable. Once the pH was stable, the flask was inoculated with an active acclimatized culture **of** *T.* ***ferrooxidans*** prepared as described above. The weight of flask with its contents (without the cotton plug) was taken initially to be able to monitor weight loss due to evaporation. The flask was placed on a laboratory bench at room temperature with adequate ventilation. The flask was manually shakened every determination. Prior to each measurement, the flask and contents (without plug) were weighed and deionized water was added to replace loss by evaporation. Around 1 mL aliquot was obtained and centrifuged at 1200 rpm for 10 min to separate solid from the supernatant. The supernatant was removed with a pipet and transferred to another clean 15 mL centrifuge tube, diluted to ***5*** mL with deionized water, acidified to pH<2 with –0.05 mL conc HNO,, and stored at 4°C while waiting to be analyzed. Meanwhile 1mL deionized water was added to all the flasks to replace the 1 mL aliquot sample.

When oxidative/bacterial activity had ceased as observed from the microscope and a stable pH has formed, the test was terminated. If the pH was below 3.5 and metals in the leachate were above regulatory limits, the sample is classified as having acid mine drainage potential or potential for bioleaching treatment. This test was completed within 23 days following inoculation.

Monitoring and Sampling

Every **3** days, the following parameters were monitored : color of solution, pH (Corning pH meter model 7), Eh (Fisher Accumet pH/Eh meter model 810) **,** bacterial growth, motility and density (MEF3 Reichert-Jung Microscope with Image analysis Hitachi KP-M1U CCD Camera at 800x magnification), and dissolved metals (inductively coupled plasma spectrometry). Dissolved oxygen (ORION oxygen meter model **860)** was also measured randomly in the solution to see if adequate oxygen was available for oxidation (oxygen solubility at 23 °C is 8.5 mg/L from the ORION oxygen probe manual). The pH meter was calibrated with pH **4** and 7 standards and all Eh readings were verified with ZoBell's solution (APHA, 1992). Utmost care was taken to avoid contamination among the replicates from the various meter probes. Each probe was rinsed thoroughly with deionized water spray and wiped with clean paper towel before doing any measurement.

Batch Kinetic Experiments

In order to obtain kinetic information about the acidification potential of the samples, another set of experiments using the AMDP test were undertaken. To observe the effects of agitation and increased temperature on the samples, the flasks and contents were placed inside an incubatorlshaker (Lab-line Instruments) which operated continuously at 175 rpm and 35 °C. To determine the effect of sterilization, control samples were prepared whereby the flasks and dry samples were placed inside the oven at 120 °C for 1 day and afterwards covered with aluminum foil and cooled completely before use. In total, there were five simultaneous batch tests with the following designation: (A) with agitation and bacteria, inside the incubatorlshaker at 35 °C and 175 rpm, (B) stationary and with bacteria placed on laboratory bench at room temperature (23-25 °C), (C) sterile conditions : flasks similar to B but with the sample and flask sterilized at 120 °C inside oven for 1 day, (D) flasks similar to C with oven sterilized samples and flasks but without any bacteria, and (E) nonsterile conditions, unsterilized medium and sample inoculated with acclimatized bacteria. Experiments B to E were all stationary experiments at room temperature. These experiments were undertaken to determine optimum conditions for the AMD potential test.

Light Microscopy with Image Analysis

For routine bacterial monitoring, one drop (–20 µL) of sample taken at the surface layer of the solution and another drop taken near the bottom of the flask were both placed side by side on a labelled microscope slide each with a 22 x22 mm cover glass. These were examined at 800x magnification using a MEF3 Reichert-Jung Microscope with a Hitachi KP-M1U CCD Camera connected to a Sony 20" television for image enhancement and attached to a Panasonic video cassette recorder. The bacterial growth and characteristics were observed visually and qualitatively as required by the AMDP procedure and noted as very high, high, medium, or low to describe density and slow, fast, and very fast for motility. A video of the bacteria at various stages of their growth as seen through the microscope was recorded showing their motility and density.

For bacterial count, one drop (–20 µL) aliquot was placed on a microscope slide with a 22 x 22 mm cover glass and examined under a light microscope at 1000x magnification. Three to four fields per sample were photographed and stored in computer format **as** an image file using an Olympus Vanox C-35 camera with a CCD X-77 video camera attached to a Macintosh Quadra 650 computer with an Image Scion 1.51 software. Direct bacteria cells from the images were counted and the total cell count/mL was calculated using a factor.

RESULTS AND DISCUSSION

The progress of oxidation was observed visually via the change of color of the solution from light grey to yellow orange which indicates ferrous to ferric ion oxidation. Oxygen level in the flasks was 5-7 mg/L through the experiment. After 23 days of bioleaching using the AMDP test, Pb was not found in the leachate in any sample or kinetic experiments. For the Philippine scale (PSC) and sludge (PSL), less than 2% leached out for both Cu and Zn with or without bacteria. On the other hand, for the Mexican scale (MSC), almost 100% of Cu and Zn were released in the leachate both for agitated and stationary experiments, respectively. Since Pb is the regulated element (and **Cu** and Zn are not), all the samples pass the regulatory requirement for leachate quality criteria (USEPA, 1990) and therefore should not be classified as hazardous wastes. Also, the leachate quality of the Philippine scale and sludge were within the limits for disposal to agricultural lands (Tyagi and Couillard, 1989) while Mexican scale requires special attention.

Only the Mexican scale has a confirmed acid mine drainage potential for **Cu** and Zn but not for Pb as shown in Figure 1. In spite of the acid generation which was supposed to promote breakdown of the sulfide mineral lattice hence more dissolution of metals, the bioleachate only contained the soluble metal sulfates of Cu and Zn and not the insoluble lead sulfate.

Further investigation using kinetic test confirmed that they all had acidification potential since the final pH of all the solutions was below **3.5** as illustrated in Figure 2. Due to space limitation, some of the results will be discussed without accompanying figures. There was no marked difference in the overall leaching of Cu, Zn, and Pb under both sterile and nonsterile conditions for all the samples tested. The contribution of bacterial effect to the overall leaching of Cu, Zn, and Pb was found more significant in the Mexican scale than the Philippine scale and sludge. This could be due to the presence of these metals as sulfides in the Mexican scale which are amenable to oxidation catalyzed by *T. ferrooxidans*.

Pb was not found in solution since it could have precipitated immediately as PbSO, and associated with the other precipitates such as jarosite, ferric sulfates and oxyhydroxides. Several researchers (Silver and Torma, 1974; Rossi, 1990) detected lead sulfate (anglesite) **through** X-ray diffraction analysis of the insoluble residual matter from bioleaching of galena or lead sulfide concentrate containing **42%** Pb and 30% **S.** Two methods as described also in the Introduction can produce lead sulfate. In the presence of ferric ion, the oxidation can be expressed by Equation *5* as follows

$$PbS + Fe_2(SO_4)_3 \dashrightarrow PbSO, + 2FeSO_4 + S^0 \qquad (5)$$

While with direct biological mediation, lead sulfate can be formed via Equation **6,** with the *T. ferrooxidans* also involved in the oxidation of elemental sulfur and ferrous ion generated in Equation *5.*

$$PbS + 2O_2 \xrightarrow{\text{bacteria}} PbSO, \qquad (6)$$

The low metal leaching in Philippine scale and sludge can be due to a number of reasons. Firstly, Mexican scale has a much higher metal content, identified as metal sulfides by X-ray diffraction (Peralta et al, 1996c) than in **PSC** and PSL. Secondly, the heavy metals in PSC and PSL may not be in sulfide forms and may not be amenable to bioleaching. Thirdly, as shown in the photomicrographs (Peralta et al, 1996a), they were not accessible to the bacteria and to the leach solution since they were bound by strongly cemented silicates.

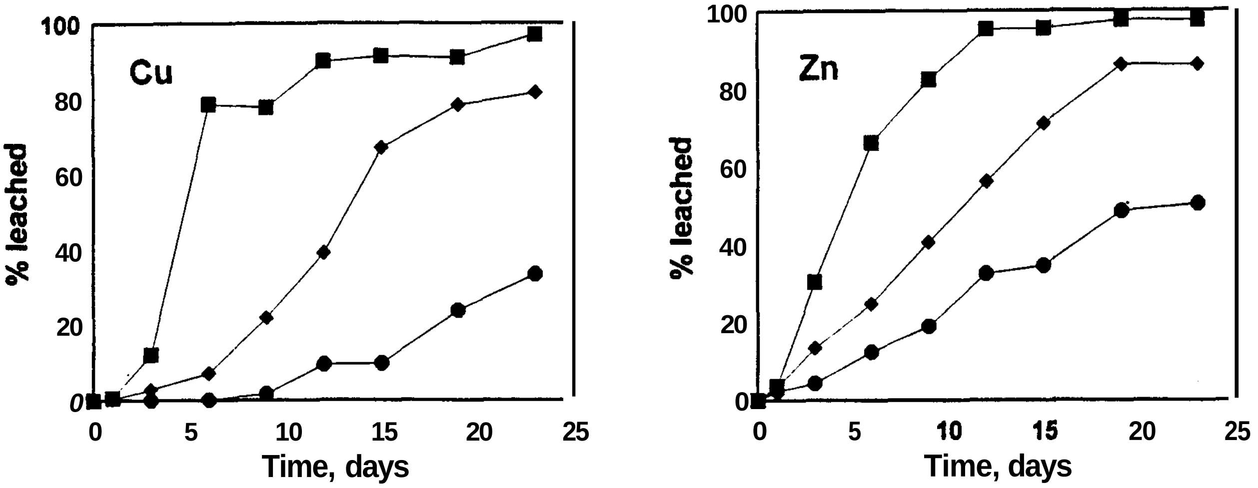

Fig 1 Bioleaching of Cu and Zn in the Mexican scale. The agitated (35 °C) and stationary (25 °C) experiments were inoculated with bacteria. Control (without bacteria) experiments were performed at 25 °C. Note also the temperature effect on metal leaching. ■ **Agitated** ◆ **Stationary** ● **Control**

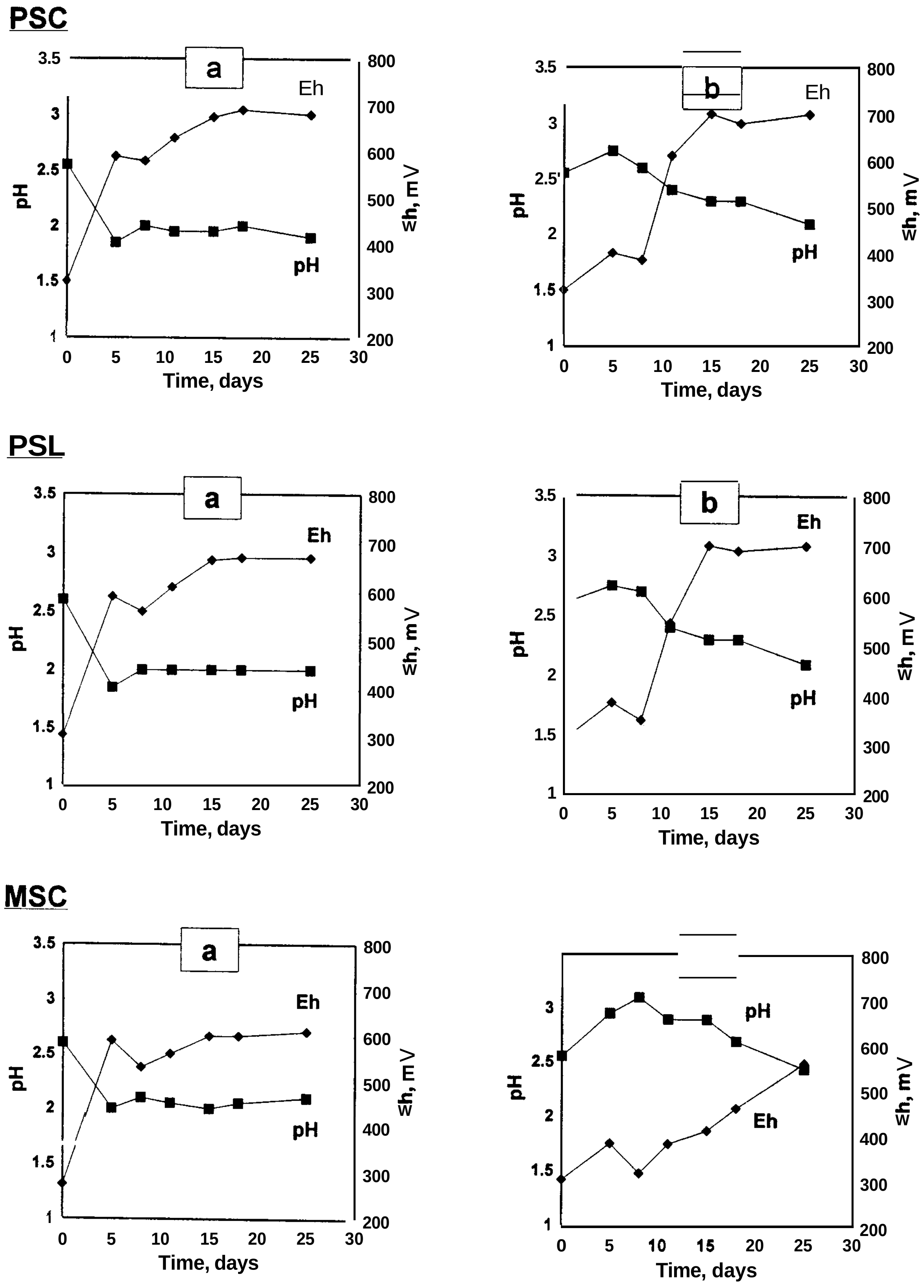

Figure 2 These graphs show the inverse relationship of pH and Eh change over time for the Philippine scale and sludge and Mexican scale. The graphs on the left column (a's) were agitated experiments at 35 ^{0}C while right column (b's) were stationary experiments at 25 ^{0}C.

EVALUATION OF THE AMDP PROCEDURE

Several criteria were used to evaluate laboratory tests to predict acid mine drainage potential. Most common evaluation criteria were simplicity, time required, equipment required, cost, ease of interpretation, and correlation with field data (Lawrence et al, 1989; USEPA, 1994). The AMDP procedure developed at the University of Toronto for geothermal residues can be described as low cost, simple, low technology, reproducible, requires little operator training, no specialized equipment needed, tolerant of sample variations, and can be performed in a nonsterile environment. The AMDP procedure has the versatility to be used more widely in laboratories with limited equipment, in actual mine/wellfield sites, and especially in developing countries where use of geothermal energy was rapidly increasing.

The main shortcomings of the BC Research Confirmation Test in relation to geothermal residues were: a) the pulp density was too high; b) no indication of redox reactions; c) no indication of bacterial viability; d) no FeSO, in the leaching medium.

CONCLUSIONS

1. ***T.ferrooxiduns*** can be cultured and acclimatized without agitation in nonsterile laboratory conditions.

2. Of the geothermal samples, only the Mexican scale was found to have acid mine drainage potential and leaching of Cu and Zn was observed. The galena likely was attacked but Pb was not found in the leachate at the end of the test and could have precipitated as insoluble PbSO,. This may also indicate that Cu and Zn can be reclaimed through microbial leaching as in mineral recovery of metals.

3. None of the geothermal residues tested in this work should have significant environmental impact in a landfill even with biological mediation.

PR TUDIES

1. Use of acid mine drainage potential test with a mixed culture of *T.* ***ferrooxidans*** and *T.* ***thiooxidans.***

2. Verification of the AMDP prediction through comparison with occurrence of acid mine drainage in geothermal stockpile sites.

3. More sampling and analysis to reflect temporal and spatial variation in characteristics of geothermal residues.

SUMMARY

Geothermal residues have sulfur content in the form of metal sulfides that indicate a potential in the long-term for acid mine drainage (AMD). AMD is experienced in coal and metal mines whereby pyrites and other sulfide minerals are oxidized releasing acidity and metals to the environment. This is usually promoted by the presence of iron and sulfur oxidizing bacteria such as ***T.ferrooxidans.***

For the geothermal residues, a new test called Acid Mine Drainage Potential test (AMDP), was developed based on an existing procedure from the mining industry. The bacteria had to be acclimatized to this new substrate (geothermal wastes) and ferrous ion had to be added to the culture media since the residues are low in Fe and S relative to mine tailings. From this test, only the Mexican scale was found to have a slight AMD potential since Cu and Zn leached out but not the regulated element Pb. Although less than 4% of Pb was in the leachate at the beginning, its disappearance was probably due to precipitation as insoluble PbSO,. Since the mineral sulfides were inside a silicate matrix and were relatively inaccessible to the bacteria, it was likely that enhanced leaching occurred primarily via indirect attack with ferric ion as the oxidant. More leaching was observed in the presence of bacteria.

The results indicate that the geothermal residues will probably not pose a direct threat to the environment but will presumably create a nuisance or aesthetic pollution due to the formation of iron precipitates which are visible as rusty orange brown solids in dumps or rivers. But since the geothermal residues have less iron content than mine tailings, there will likely be less iron precipitation compared to coal or metal mines.

From the results of this work, it is likely that the geothermal residues can be safely disposed in a landfill environment.

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