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METAL CONCENTRATIONS AND CONCOMITANT METAL MOBILITY IN UNSATURATED MINE AND MILL WASTES

by
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ABSTRACT

The shallow sediments in the Smelterville Flats portion of the Coeur d'Alene River Valley in north Idaho consist of a heterogeneous mixture of mine wastes and alluvium reworked during flood events. The wastes cover an area of approximately eight square kilometres. Atomic Absorption Spectroscopy and Inductively-Coupled Plasma analyses on mine waste-sediment samples collected to the depth of the water table show maximum metal concentrations of about 20% Fe, 12% Pb, 10% Zn, 3% Mn, 2.8% Al, 0.13% Cu, 0.1% Cd, 370 ppm As, 170 ppm Ag, 150 ppm Sb, and 70 ppm Ni. The positive correlation between Fe, Ca, and Mg concentrations and sample pH suggests that the carbonate gangue minerals (siderite, ferrodolomite, and ankerite) control the pH. Other metals that exhibit a positive correlation with sample pH also may be present in the carbonate minerals. Identification of factors that govern metal mobility suggests that : (a) the pH of the water in the uppermost aquifer plays a minor role in controlling the solubility of most metals in the solid waste samples; b) the carbonate gangue minerals buffer the ground water pH and hence the solubility of metal sulfides; c) solubility of the carbonate gangue minerals decreases under anaerobic conditions, thereby allowing the ground water pH to decrease; d) solubility of Pb, Zn, and Cd correlates directly to anaerobic conditions as opposed to aerobic conditions.

INTRODUCTION

This study constitutes and effort to characterize geochemically the uncontrolled mine and mill wastes (hereinafter called waste-sediment) at the Smelterville Flats area, near Kellogg, Idaho (Figure 1). The concentrations and mobility of the metals in the waste-

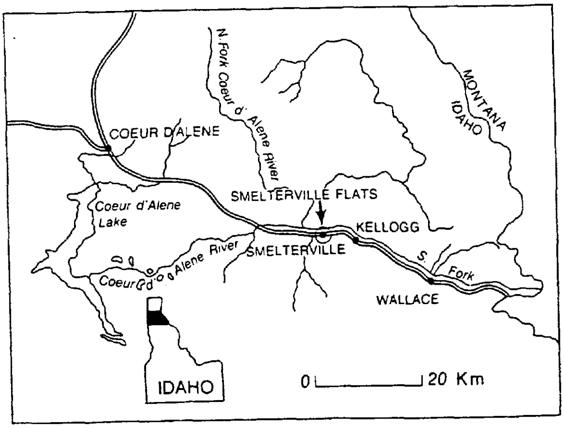

Figure 1. Location map for the Smelterville Flats, Kellogg, Idaho (Adams, 1989)

sediment mixture is the principal concern in this study. This information is necessary for formulating and evaluating alternative procedures for mitigating surface water and ground water contamination at the site. The study is part of a larger project entitled, "Evaluation of Waste and Water Contamination for Closure of a Hard Rock Mine Complex", funded by cooperative agreement number C0278001 between the United States Bureau of Mines and the College of Mines and Earth Resources at the University of Idaho.

Mining operations within the Coeur d'Alene Mining District in north Idaho have continued for nearly a century. Degradation of water resources resulting from mining and milling activities along the South Fork of the Coeur d'Alene River has been documented since 1940 (Ellis, 1940). Recent water quality regulations as well as advanced milling and mining techniques have combined to diminish significantly the metal concentrations and high stream sediment loads during the last decade. However, controlled and uncontrolled mine waste depositional sites not associated with currently active mining operations still remain.

DESCRIPTION OF AREA

Smelterville Flats constitutes an area of about eight square kilometres in the flood plain of the South fork of the Coeur d'Alene River. It is located a few kilometres west of Kellogg, Idaho, and approximately 50 km east of Coeur d'Alene (Figure 1). Unconsolidated sediments of glacial and fluvial origin overlie a bedrock of Middle Proterozoic metasediments of the Belt Supergroup. The unconsolidated sediments above the bedrock at Smelterville Flats consist of three hydrostratigraphic units. These units are an upper and a lower aquifer, separated by an aquitard. The Smelterville Flats area has received, over the years, wastes from many upstream mining and milling operations. Mines began to discharge wastes into the South Fork of the Coeur D'Alene River in the 1880s; this practice continued for more than eighty years until the late 1960s. A small dam built across the South Fork of the Coeur d'Alene River in 1901 (and breached by a flood in 1932/33) deposited mine and mill waste atop the upper aquifer on Smelterville Flats (Ioannou, 1979), where they still remain.

SAMPLING AND ANALYSES

Waste-sediment samples were collected in six trenches excavated to the water table beneath Smelterville Flats. The locations of the six trenches (labelled T1 through T6) are shown on Figure 2. Samples were collected at one foot vertical intervals from the surface to the water table, which ranged between 6 and 9 feet in depth. After drying, the samples were split into three equal aliquots. One of the aliquots was used for pH determination, another for grain size distribution and metal concentrations, and the third for laboratory batch tests. Each sample was sieved for 30 minutes through meshes of 0.053, 0.212, 0.50, and 2.00 mm, thereby producing five fractions consisting of silt and clay, fine sand, medium sand, coarse sand, and granule. The weight percent of each fraction along with additional details is presented in Towatana (1990). Preliminary tests showed that in all the trenches the fine fractions had the highest metal concentrations. Therefore, only the three smallest size fractions were analyzed for metal content. EPA method 3050 (EPA, 1986) was used for determination of metal concentration. Preparation blanks (double deionized water and reagents) were carried throughout the sample preparation and analytical process. The method of Mclean (1982) was followed during the measurement of waste-sediment pH.

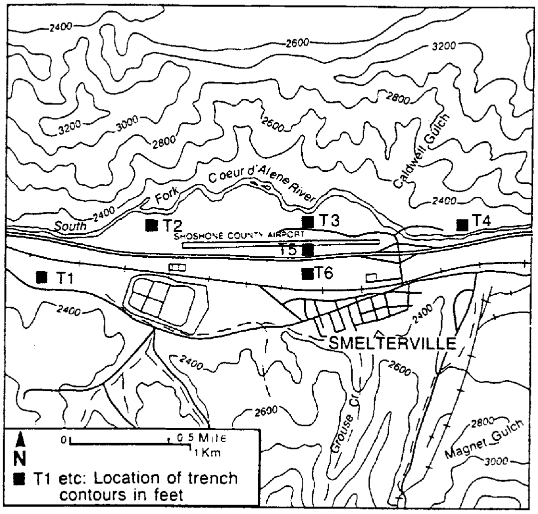

Figure 2. Map of the Smelterville Flats area, showing the location of the trenches from which samples were derived.

pH AND METAL CONCENTRATIONS

The data on pH and metal concentrations in the waste sediment samples from the six trenches are shown in Towatana (1990) and summarized in Figures 3 and 4. Comparison between average crustal values from Krauskopf (1979) and data presented in Figure 4 reveals that concentrations of heavy metals in the waste-sediment mixture vary from 10 to 10,000 times the crustal average. Al and Ni concentrations are below crustal average, thereby reflecting their paucity in the mining and milling wastes. The concentration of Al at approximately an order of magnitude less than the crustal average suggests that the presence of aluminum-bearing clay minerals on the Flats is very limited. As shown by data presented in tabular form in Towatana (1990) and summarized in Figures 3 and 4, a positive correlation exists between waste-sediment pH and metal concentration, with Fe, Ca, and Mg showing especially strong positive correlations (R=0.85 and 0.73). We attribute this relationship to pH buffering by carbonate gangue minerals. The carbonate minerals siderite, ferrodolomite, and ankerite have been reported in the mineralized veins of the Coeur d'Alene Mining District (Fryklund, 1964; Reece, 1974; Ralston et al., 1973). The following reactions may be occurring:

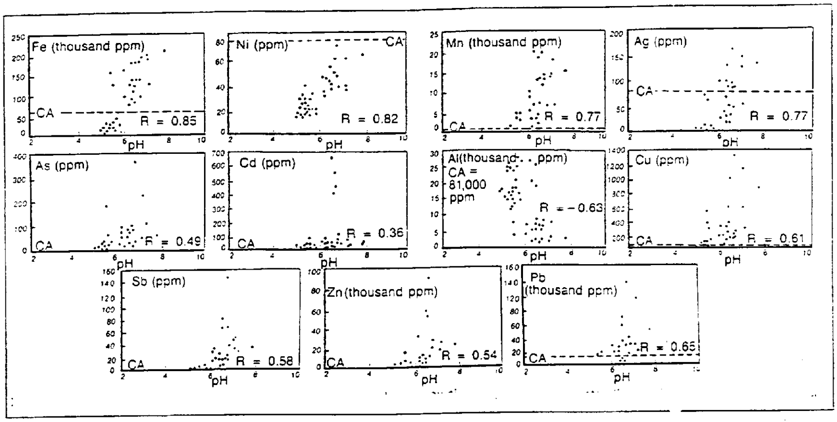

Figure 3. Waste-sediment pH versus metal concentrations (silt and clay size fractions). CA indicates the crustal average for each metal (from Krauskopf, 1979).

(7) $FeCO_3^- + H^+ = Fe_2^+ + HCO_3^-$ (Dissolution)
(8) $HCO_3 + H^+ = CO_2 + H_2O$ (Hydrolysis)
(9) $Fe^{2+} + 0.25\ O_2 + H^+ = Fe^{3+} + 0.5\ H_2O$ (Oxidation)
(10) $Fe^{3+} + H_2O = Fe(OH)_2^- + H^+$
(11) $Fe(OH)_2 + H_2O = Fe(OH)_2^- + H^+$
(12) $Fe(OH)_2 + H_2O = Fe(OH)_3 + H^+$

Siderite dissolves in the manner given in reaction 7. Dissolution, hydrolysis and oxidation of one mole of siderite consumes three hydrogen ions as shown in reactions 7, 8, and 9 respectively. However, hydrolysis of a ferric ion to ferric hydroxide liberates three hydrogen ions (reactions 10, 11, and 12). If all the reactions went to completion, the net effect of siderite on pH would be neutral. However, the rate of chemical oxidation of iron is relatively slow and pH dependent, whereas the rates for the hydrolysis reactions are much faster. Reactions 10 through 12 show that the rate of hydrolysis of ferric ions is pH dependent, which suggests that the neutralizing effect of siderite will vary with the conditions under which it is dissolved. Furthermore, substitution of Ca_2^+ Mg_2^- for Fe_2^- in siderite produces higher pH values. Reece (1974) obtained pH values of 7.1-8.4 after 15 minutes using 1 cc of siderite crushed to 0.02 mm and 20 ml of distilled water. Dolomite yielded pH values of 7.6-9.8 under the same conditions. Carbonate gangue minerals apparently are associated with heavy metals in the district. Reece (1974) also studied samples from the South Fork of the Coeur d'Alene River. He reported that all the samples that contained high concentrations of Pb, Zn, and Cd consisted mainly of the minerals siderite and quartz (with some magnetite). Reece concluded that the intensity of the x-ray diffraction pattern for siderite correlated with the concentration of heavy metals in waste-sediment samples. The higher the x-ray peaks for siderite, the higher the concentrations of Fe, Zn, Pb, Mn, Cu, and Cd in the samples.

Galbraith and others (1972) observed a similar correlation between concentrations of metals and pH of tailings on nearby Cataldo Mission Flats. They observed a distinct rise in the concentrations of Zn, Pb, Mn, Fe, Cu, Ag, and Mg at pH 6.6 or above. They concluded that this increase in metal concentration reflected sulfate reducing bacteria activity. However, such sulfate reducing bacteria generally thrive at depth and under anaerobic conditions, whereas these zones of high metal concentration that showed high tailings pH are located at shallow depth (1-3 feet). They observed a decrease in both metal concentrations and tailings pH with increasing depth. This relationship between metal concentration and pH of the tailings at Cataldo Mission Flats might also be explained by the buffering action of carbonate gangue minerals.

Identification of the relationship between pH and metal concentration may constitute a useful parameter for an approximate mapping of the metal rich portion of the waste sediment mixture at Smelterville Flats. A distinctive rise in concentrations of Fe, Ag, Pb, Ni, Cd, Sb, Zn, and Cu was observed at pH 6.0 (waste:water = 1:2 by weight). Waste-sediment samples with pH values of 6 or above are likely to contain high metal concentrations which are suitable for reprocessing.

BATCH TESTS

Batch tests performed in this study were designed to identify the important factors that control the solubility and mobility of the toxic metals in the upper aquifer. Each sample chosen for the batch tests was the one with the highest metal concentrations.

Groundwater pH, oxidation potential (Eh), waste:water ratio, and residence time were the variables tested. Geochemical conditions in the upper aquifer at Smelterville were simulated as closely as possible. The pH of the text solution was adjusted (using H_2SO_4 and NaOH to 4.7, 5.7, and 7.2, which are equal to the minimum, average, and maximum pH values determined on groundwater in the upper aquifer (Kunkel, 1990). The oxidation potentials of two different environments in the upper aquifer were simulated. The first condition was anaerobic and continuously wet and intended to simulate conditions in the saturated zone. The second condition was aerobic, alternately wet and dry, intended to simulate the zone of a fluctuating water table, or conditions in the unsaturated zone subject to periodic precipitation. The effects of varying the waste to water ratio were studied by running two parallel anaerobic tests, one with a waste to water ratio of 1: 4 by weight, and the other with 1: 20 by weight. The ratios chosen were best estimates of the extreme values that actually occur in the upper aquifer. The residence times for the tests were limited to 600 hours under anaerobic conditions and 360 hours under aerobic conditions. Previous studies (Williams et al., 1979) had shown that equilibrium was attained after 300-400 hours.

For the aerobic tests, 10 g of waste-sediment sample were mixed with 40 g of solution of selected pH, stirred for 90 minutes and then allowed to stand for 90 minutes. The sample was then filtered and the filtrate analyzed for pH and metal content. After drying the residue for 72 hours, another 40 g of solution (of the same pH) was added and the procedure repeated. The samples were treated a total of six times, with a 72 hour drying period between each analysis.

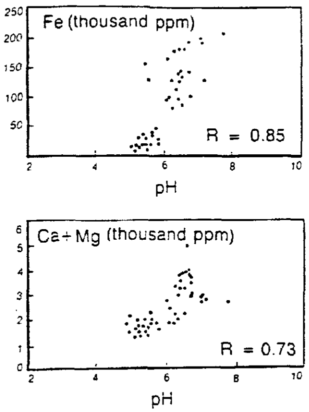

Figure 4. Waste-sediment pH versus Fe, Ca and Mg concentrations.

The anaerobic tests included two different waste to water ratios. Either 10 g of sample were mixed with 40 g of solution, or 5 g of sample were mixed with 100 g of solution.The samples then remained submerged and undisturbed for 12, 24, 96, 336, or 600 hours. After the predetermined residence time, each sample was stirred for 45 minutes, allowed to stand for 60 minutes and then filtered. The filtrate was then analyzed.The concentrations of the following metals were measured: Ag, As, Ca, Cd, Cu, Fe, Mg, Mn, Pb, Sn, and Zn, along with pH.

RESULTS OF BATCH TESTS

Results of the batch tests are shown in Figures 5 through 15, where metal concentrations in solution are plotted against residence times. Initial concentrations of each metal in the waste-sediment mixture are also given. Tests were conducted at three different pH values.

Figures 5-15 show that the solubility of most of the metals was almost the same for the three different initial pH values. In Figure 16, the pH of the solution is plotted against residence time for each of the initial pH values; Figure 16 shows that the variation in the solution pH is very small compared with the variation in the initial pH. Under aerobic conditions initial pH values of 4.7, 5.65, and 7.2 had all stabilized at pH 6.2 after three hours. PH then increased gradually with time, stabilizing to about 6.8 after about 200 hours. Under anaerobic conditions, the pH of the solution stabilized to just over 6 after a few hours, but then decreased with the increasing residence time, approaching a pH of 5 (and still decreasing) after 600 hours. The ratio of waste to water had little effect on the pH, except that the rate of decrease of pH was very slightly greater in the samples with a higher water to waste ratio. These observations suggest that the pH was buffered by some component of the waste. The most likely candidates are the gangue carbonates, siderite, calcite, dolomite, and ankerite. This suggestion is strengthened by the observations on the behavior of Ca, Mg, Fe, and Mn in the batch tests. Ca, Mg, Fe,and Mn are far more soluble under aerobic conditions than under anaerobic conditions (Figures 5-8).

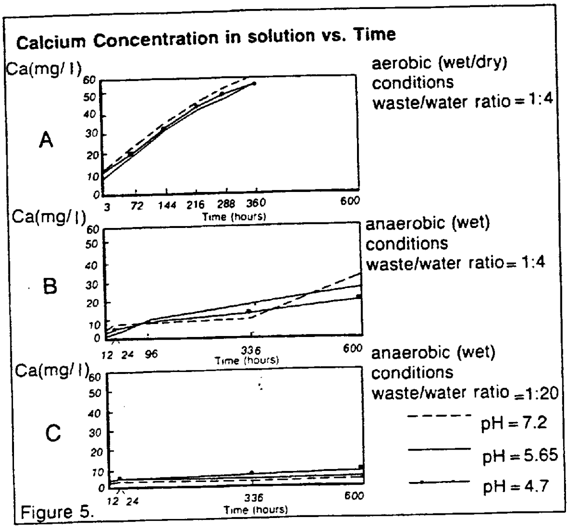

Figure 5 Calcium concentration in solution versus time. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight.

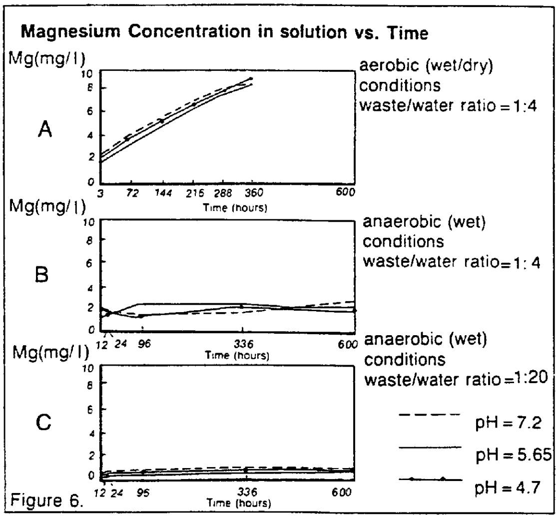

Figure 6. Magnesium concentration in solution versus time. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

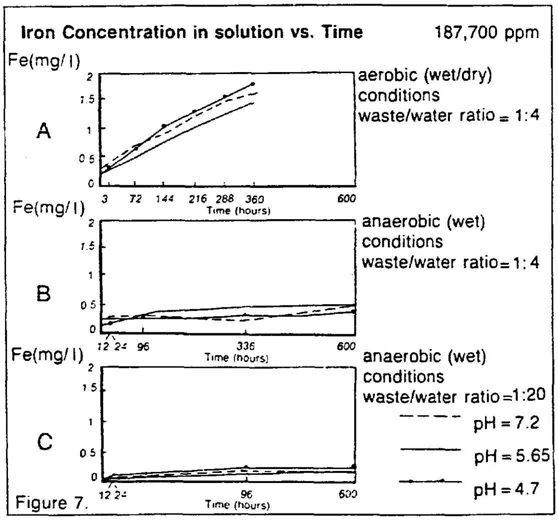

Figure 7. Iron concentration in solution versus time. Original concentration of Fe in waste-sediment = 197,700 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

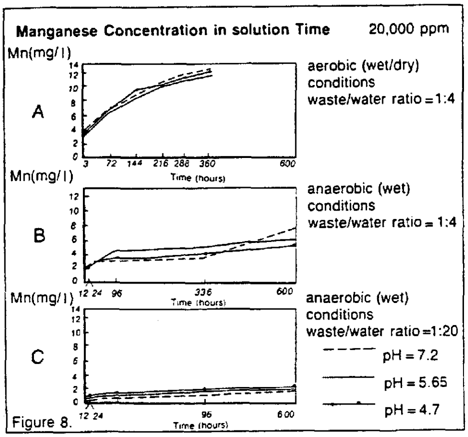

Figure 8. Manganese concentration in solution versus time. Original concentration of Mn in waste-sediment = 20,000 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

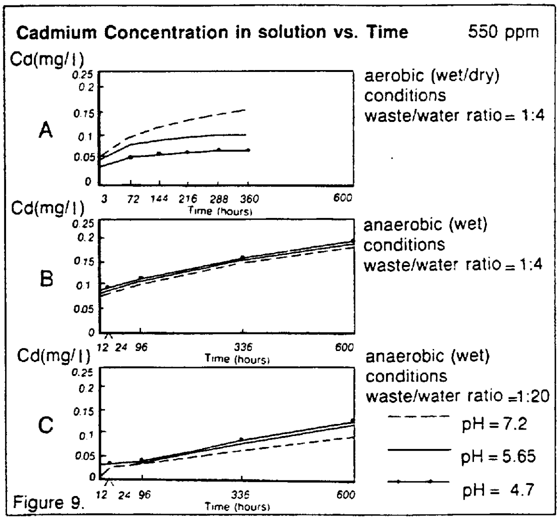

Figure 9. Cadmium concentration in solution versus time. Original concentration of Cd in waste-sediment = 550 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

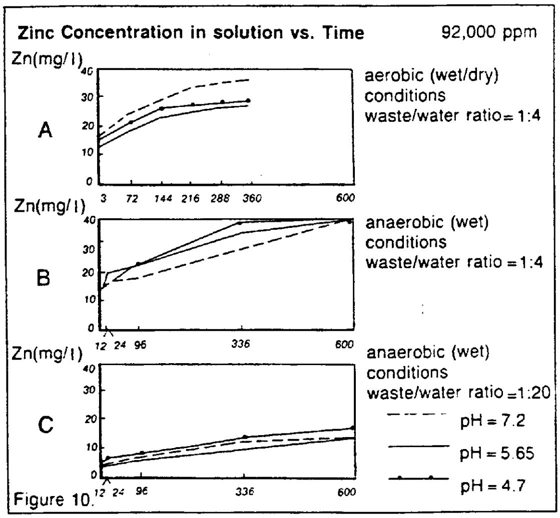

Figure 10. Zinc concentration in solution versus time. Original concentration of Zn in waste-sediment = 92,000 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

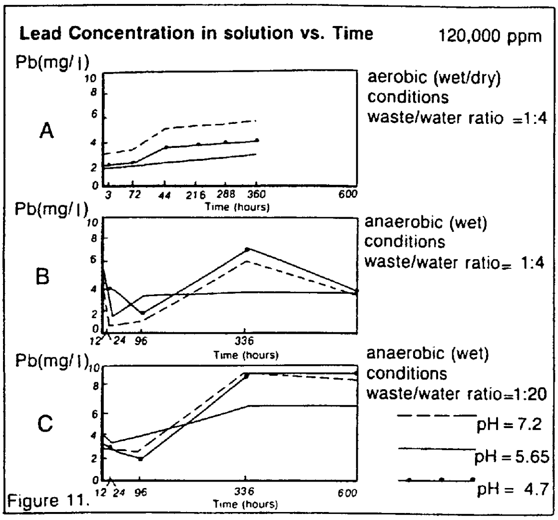

Figure 11. Lead concentration in solution versus time. Original concentration of Pb in waste-sediment = 120, 000 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight c) anaerobic conditions with waste:water ratio = 1:20 by weight

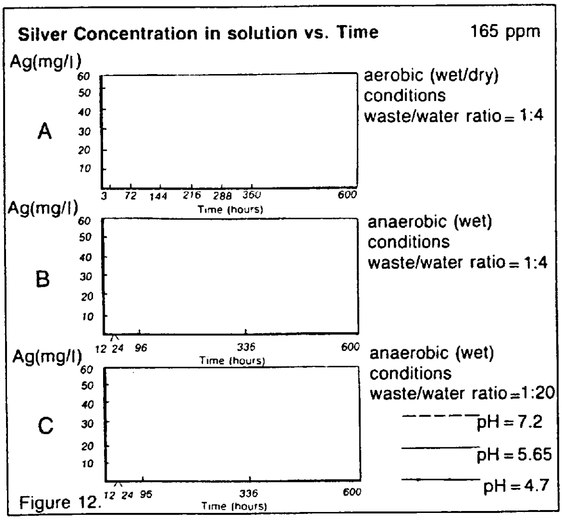

Figure 12. Silver concentration in solution versus time. Original concentration of Ag in waste-sediment = 165 ppm.
(a) aerobic conditions with waste:water ratio = 1:4 by weight
(b) anaerobic conditions with waste:water ratio = 1:4 by weight
(c) anaerobic conditions with waste:water ratio = 1: 20 by weight

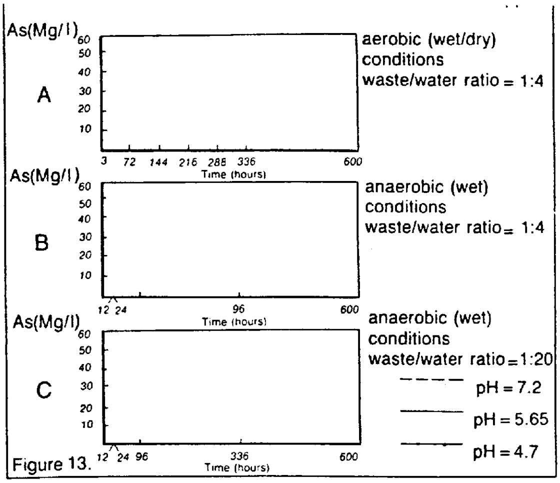

Figure 13.

Figure 13. Arsenic concentration in solution versus time. Original concentration of As in waste-sediment = 93 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

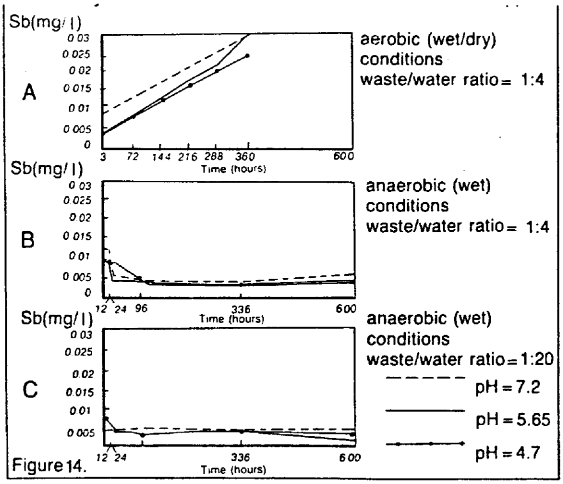

Figure 14.

Figure 14. Antimony concentration in solution versus time. Original concentration of Sb in waste-sediment = 154 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1: 20 by weight

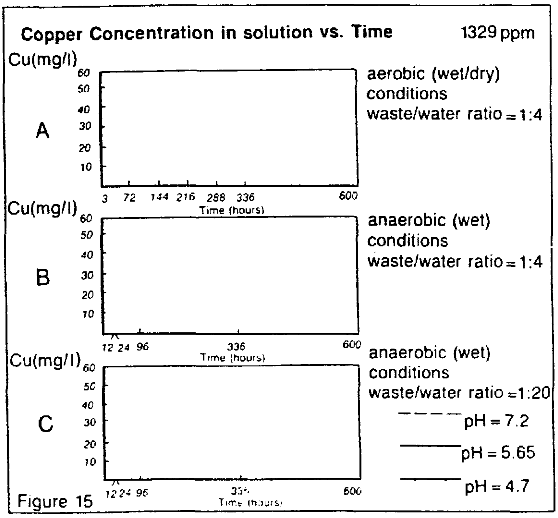

Figure 15. Copper concentration in solution versus time. Original concentration of Cu in waste-sediment = 1329 ppm. (a) aerobic conditions with waste:water ratio = 1:4 by weight (b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

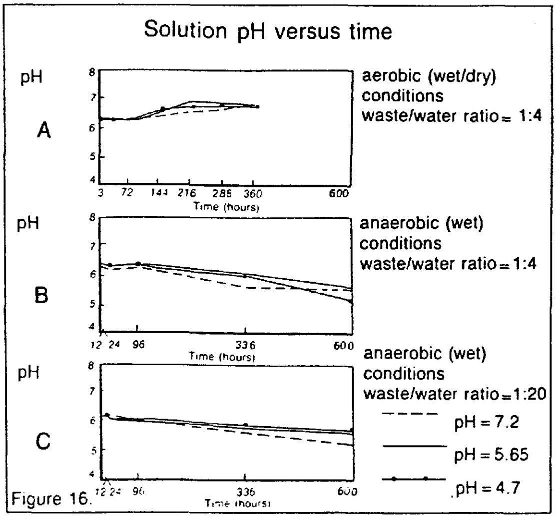

Figure 16 . Solution pH versus time.(a) aerobic conditions with waste:water ratio = 1:4 by weight(b) anaerobic conditions with waste:water ratio = 1:4 by weight (c) anaerobic conditions with waste:water ratio = 1:20 by weight

Under aerobic conditions all these metals increased in concentration by a factor of 3 or more (4 for Mg, 5 for Ca) after 360 hours. Under anaerobic conditions, concentrations had increased very little after 360 hours (negligible for Mg and Fe but by a factor of 2 or less for Ca and Mn). The greater solubility of the carbonates under aerobic conditions probably causes the greater buffering of low pH groundwater under aerobic conditions (Figure 16). The result of this buffering appears to be a decrease in the mobility of the toxic metals (Cd, Zn, and Pb). Figures 9, 10, and 11 show that the concentrations of these metals increase only slightly (by a factor of 2 or less) under aerobic conditions in groundwater of pH 4.7, and tend to stabilize after about 150 hours. Under anaerobic conditions (where the buffering capacity of the carbonate gangue minerals appears to be decreased), the concentrations of Cd and Zn have reached the maximum aerobic values after about 150 hours; these concentrations continue to increase and were still increasing after 600 hours. The solubility of Pb under anaerobic conditions is more complex, perhaps due to complexing of the Pb atom into other molecules (Lindsay, 1972). Maximum concentrations again are greater than those under aerobic conditions.

Ag and As were not detected in any of the solutions, reflecting their relative insolubility under all test conditions (Figures 12 and 13). Detection limits for these metals are ppb on the ICP/MS. These metals should constitute no problem with respect to groundwater quality in samples similar to those at a depth of 6 feet in trench 1. Cu was detected only under anaerobic conditions at pH 4.7. Concentrations increased from 0.02 ppm to 0.04 ppm compared with an initial concentration of 1329 ppm in the waste-sediment mixture. Sb is far more soluble under aerobic conditions. Figure 14a shows concentrations of 0.02 ppm to 0.02 ppm after 360 hours (on a steeply increasing trend), 0.005 ppm and stable under anaerobic conditions (Figures 14b and c), compared to an initial concentration of 154 ppm in the waste-sediment. The most soluble metals were Zn, Cd, Mn, and Sb; their concentrations in solution increased by at least an order of magnitude more than those of the other metals, compared with their initial concentrations in the waste-sediment.

CONCLUSIONS

The mine and mill wastes that are distributed on Smelterville Flats are mixed heterogeneously with alluvium. Analysis of samples collected from six separate backhoe trenches excavated to the dry weather water table at Smelterville Flats show a positive correlation of waste-sediment pH with total concentrations of Ag, As, Ca, Cd, Cu, Fe, Mg, Mn, Ni, Pb, Sb, and Zn. Carbonate gangue minerals present in the wastes are considered to cause this relationship. Increasing waste-sediment pH with increasing Fe, Ca, and Mg concentrations in the wastes along with an abundance of gangue siderite, ferrodolomite, and ankerite and the association of carbonates with high metal concentrations in wastes sampled from the South Fork of the Coeur d'Alene River (Reece, 1974) all suggest that carbonate gangue minerals are an important geochemical factor in the waste sediments. Recognition of the relationship between waste sediment pH and metal concentration means that pH measurements conceivably could be used as a "pathfinder" to locate those wastes with greatest potential for reprocessing. Batch experiments, designed to investigate the parameters (pH, Eh, waste:water ratios, and time) that govern the solubilities of the metals, indicate that the pH of water in the upper aquifer apparently plays only a minor role in controlling the solubility of the metal ions. The waste-sediment samples from trench l used in the batch tests have a buffering effect on the pH of the solution. Higher acidity (low pH) and greater solubility of Pb, Zn, and Cd were observed to correlated directly to anaerobic conditions as opposed to aerobic conditions. These batch tests also showed that the solubility of Pb is complex. It produces fluctuations in Pb concentrations and solubility trends and is reverse to some trends observed for Zn and Cd. An absence of ferrous-ferric oxidation in the crystal lattices of the carbonate gangue minerals under anaerobic conditions decreases the gangue mineral solubility. The reduced solubility of gangue minerals may decrease the pH of solutions, thereby enhancing the solubility of Zn, Cd, and Pb in the wastes. Thus, flooding of the Smelterville mine and mill wastes (anaerobic conditions) may enhance the solubility of Pb, Zn, and Cd currently held in the wastes. Zn, Cd, Sb, and Mn are the most soluble metals tested, Sb being particularly soluble under aerobic conditions.

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