THE REMOVAL OF NICKEL FROM MINE WATERS USING BACTERIAL SULFATE REDUCTION[1]

by

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Abstract. Experiments were done to determine if a mushroom compost-based sulfate reduction system could be used to treat nickel-contaminated mine waters. Sulfate reduction systems were established in columns containing acid-washed mushroom compost. Simulated mine waters containing 2000 mg/L sulfate and 50 to 1000 mg/L nickel were adjusted to pH 4.5 and pumped through the columns at flow rates between 15 and 25 mL/hr. During the first nine days of operation, virtually all influent nickel was removed in the columns by sorption and ion exchange mechanisms. The nickel removal rate then dropped to 18 to 30 mg Ni/day (7.8 to 12.8 nmol/g-substrate/day), probably due to low sulfate reduction rates. When sodium lactate was added to the inflow, a sustained and seven-fold increase in the nickel removal rate was observed. Bacterial sulfate reduction rates measured in mushroom compost-based wetlands exposed to coal mine drainage are 2 to 20 times faster than the rates observed in this study. Low sulfate reduction rates in the columns may have been due to the inhibition of microbial activity by high nickel concentrations and the removal of labile organic compounds from the mushroom compost substrate during acid-washing. Results of this study indicate that bacterial sulfate reduction can effectively treat nickel concentrations up to 500 mg/L if labile carbon is nonlimiting.

Introduction

The sulfide-rich Duluth gabbro deposit in northeastern Minnesota is an important source of nickel, copper, zinc, cobalt, and associated precious metals (Natarajan et al. 1982). Mining of this ore body will inevitably result in the production of contaminated water. Acidic and metal-contaminated leachates now drain from waste rock removed from a test shaft driven into a highly-mineralized zone of the deposit near Babbitt, MN (A and B, Table 1). Other portions of the Duluth Complex are being disturbed by the open-pit mining of underlying taconite orebodies. Currently, this sulfide-rich waste rock is being stockpiled to permit the mining of subjacent taconite orebodies. The

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[1] Paper presented at 1991 American Society for Surface Mining and Reclamation Meeting, Durango, CO, May 14-17, 1991.

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Proceedings America Society of Mining and Reclamation, 1991 pp 97-108
DOI: 10.21000/JASMR91010097
https://doi.org/10.21000/JASMR91010097

Table 1. - Water quality[1] of leachates from Duluth Gabbro waste rock.

	Concentrations, mg/L						
Sample	pH[2]	SO_4^{2-}	Cu	Ni	Co	An	Fe
A	4.8	2740	63.0	196.0	9.1	10.3	0.10
B	4.2	3430	115.0	198.0	9.8	10.0	0.20
C	6.9	1125	0.02	1.6	0.06	0.02	0.10
D	7.2	1910	0.11	2.2	0.10	0.04	0.10
E	7.1	4000	0.64	50.0	3.4	3.6	0.10
F	5.4	1425	0.46	14.0	0.94	1.6	0.20

[1] Data provided by Kim Lapakko, Divison of Minerals, Minnesota Department of Natural Resources.

[2] Standard units.

water quality of the drainage emanating from these stockpiles exceeds water quality standards by as much as 2 to 3 orders of magnitude (samples C, D, E, and F, Table 1).

Prior to mining the Duluth Complex ore deposit, water pollution control measures must be in place to ensure the protection of the region's environment. A number of options are available for the treatment of metal-contaminated drainages. Chemical treatment plants that add alkaline materials are able to treat water effectively, but they are expensive to build, maintain, and operate. Metal adsorption onto sedge peat (Eger and Lapakko 1988) and polymeric beads containing Sphagnum peat (Jeffers 1990), are both demonstrated, low-cost techniques for treating stockpile drainages with total metal concentrations of less than 100 mg/L. However, the high total leachate metal concentrations from waste rock piles at the Babbitt test shaft (often exceeding 300 mg/L) would tend to saturate the adsorption/absorption capacity of sedge- or Sphagnum-based treatment systems relatively quickly. The required frequency of Sphagnum replacement or acid stripping (in the case of peat-containing beads) could make the use of these treatment methods economically impractical.

The precipitation of metals as insoluble sulfides is another potential treatment method for acidic Duluth gabbro leachates. Crushed limestone and gaseous H_2S have been used previously to treat acid mine waters with a high iron content (Streeter 1970). In this technique, an amorphous iron monosulfide precipitate was formed by the reaction of iron and H_2S. Crushed limestone was used to elevate the pH to about 6, where amorphous iron monosulfide was stable. Nickel, copper, and many other metal ions would both be expected to precipitate more readily than iron in the presence of excess hydrogen sulfide (Ehrlich 1981). Sodium sulfide (Na_2S) has also been added to complex wastewaters to remove Cd, Zn, Pb, and Cu as insoluble sulfides (Bhattacharyya et al. 1979). Oxyanions of selenium and arsenic were also removed by this process, presumably via the formation of elemental selenium and arsenic sulfide minerals.

One advantage of sulfide precipitation over alkali addition as a chemical treatment is that a more compact sludge is produced that can be introduced directly into conventional smelters for metal recovery. However, a sulfide precipitation plant would be expensive to operate because of high H_2S or Na_2S costs. There are also safety concerns when transporting or applying toxic H_2S gas or caustic Na_2S. The high cost of H_2S can be avoided by biologically-generating H_2S via dissimilatory sulfate reduction. Biological sulfate reduction consumes only waste organic material and sulfate, a major constituent of acid mine waters. Safety concerns would also be lessened because only the amount of H_2S necessary to precipitate metals would be generated in a properly designed system.

Dissimilatory sulfate-reducing bacteria reduce inorganic sulfate or other oxidized sulfur forms to sulfide. This sulfide is not incorporated into the organism but is released as free H_2S. Desulfovibrio and Desulfotomaculum are the two best known genera of these bacteria. They are strict anaerobes and are severely inhibited by even small amounts of oxygen. They will, however, survive long periods of oxygen exposure and become active when anaerobic conditions are restored. In fresh waters, bacterial sulfate reduction is often limited by the availability of sulfate ion (Herlihy and Mills 1985), but this is seldom the case in mine drainages resulting from the oxidation of sulfide minerals. Sulfate reduction activity has likewise been detected at environmental temperatures between 0 and 70° C (Postgate 1979).

Bacterial sulfate reduction does appear to be inhibited in acidic environments below pH 4 (Wakao et al. 1979), a common characteristic of acid mine drainage. This does not preclude the use of sulfate-reducing bacteria in acidic environments, however, because of their ability to generate alkalinity via their metabolic activity. The overall sulfate reduction process can be represented by the following generalized equation:

$$\text{Simple organic compounds} + SO_4^{2-} \xrightarrow[\text{anaerobic}]{\text{bacteria}}$$

$$\text{acetate} + H_S^- + HCO_3\text{-} \qquad (1)$$

In this reaction, the organic compounds and sulfate are converted to acetate, bisulfide or hydrogen sulfide, and bicarbonate. Application of this process in the treatment of acidic mine effluents may thus help to improve water quality in three ways:

1. sulfide generated as bisulfide or hydrogen sulfide can react quickly with metals to form insoluble precipitates and decrease dissolved metal concentrations,
2. alkalinity generated as bicarbonate and acetate can help neutralize acidity and raise effluent pH, and
3. dissolved sulfate concentrations can be reduced.

This paper presents the results of laboratory experiments designed to determine if bacterial sulfate reduction is a potential treatment technique for nickel-contaminated mine drainage.

Materials and Methods

Sulfate reduction experiments were carried out in 5.1 cm (2 in) I.D. by 45.7 cm (18 in) long columns containing 400 g of mushroom compost. Mushroom compost is a waste product of mushroom farming that consists of composted straw, hay, horse and poultry manure, ground corncobs, gypsum, and limestone. As received, this material contains an intrinsic alkalinity equal to 35 tons $CaCO_3$/1000 tons. Initially, deionized water adjusted to pH 1 with concentrated hydrochloric acid was passed through the columns to remove the intrinsic alkalinity of the compost. When the pH of the column effluents dropped below 2, this treatment was suspended. The columns were then inoculated with leachate from fresh mushroom compost and a circumneutral solution containing 2000 mg/L SO_4^{2-} (as Na_2SO_4) was passed through the columns at a 25 ml/hr flow rate for 2 weeks. After the presence of viable populations of sulfate-reducing bacteria (as growth in Postgate's medium B; McIntire et al. 1990) was demonstrated for each column, pH 4.5 solutions containing 2000 mg/L SO_4^{2-} and 50, 100, 200, 500, and 1000 mg/L Ni (as $NiCl_2 \cdot H_2O$) were passed through the columns at flow rates ranging from 15-25 mL/hr (12.5 hr average residence time).

After 20 weeks, 3500 mg/L lactate, as sodium lactate was added to the influent solutions (pH adjusted to 4.5). No effort was made to deplete the oxygen content of the influent solutions. Therefore, the dissolved oxygen content of solutions entering the columns would be in equilibrium with the atmosphere, a condition likely to be encountered at most mine sites. The effluent from each column was sampled weekly and analyzed to determine pH, acidity/alkalinity, and concentrations of Ni, SO_4^{2-}, and lactate. Nickel was measured by ICP atomic emission spectroscopy; sulfate and lactate were both measured by liquid chromatography. Acidity and alkalinity were determined by titration and expressed as mg/L $CaCO_3$ equivalent. The sulfide content of column effluents was measured by sulfide ion sensitive electrode when hydrogen sulfide odors were detected in effluent reservoirs. At the completion of the experiment, the two columns receiving 500 mg/L nickel inflow were frozen and cut into three equal segments. Acid volatile sulfide (AVS) distillations were performed on each segment (Jorgenson 1978). AVS techniques were originally developed to determine iron monosulfide accumulations in substrates undergoing sulfate reduction. The applicability of AVS techniques to other biogenically-precipitated metal sulfides is currently being evaluated.

Results and Discussion

Adsorption an Absorption Phase

For the first nine days of operation, virtually all influent nickel (fig. 1-4) and some of the sulfate (fig. 5-8) were removed by passage through the mushroom compost column. Sorption mechanisms are thought to be primarily responsible for their removal during this period. The extent that sulfate reduction may have contributed to nickel and sulfate removal during this period cannot be determined accurately. Potential indicators of bacterial sulfate reduction activity, such as increases in pH (fig. 9) and alkalinity (fig. 5-8), are masked by the effects of ion exchange that result in a lowering of pH and alkalinity. The decrease in pH from 4.5 at the inflow to about 3.7 in the effluent (fig. 9) can be attributed to Ni^{2+} displacement of H_3O^+ from ion exchange sites. Based on the

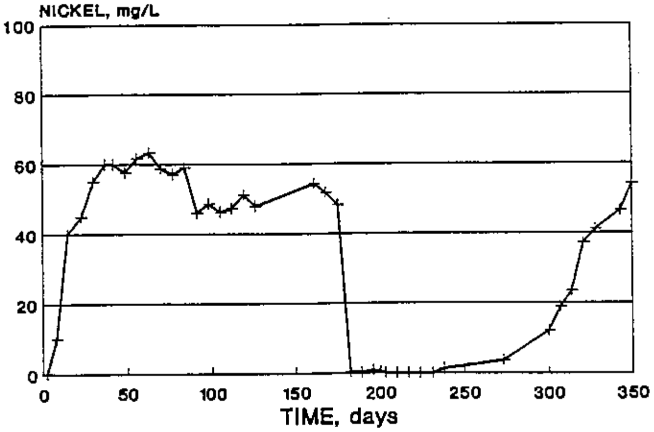

Figure 1. Average effluent nickel concentrations from two columns receiving aqueous solutions containing 100 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

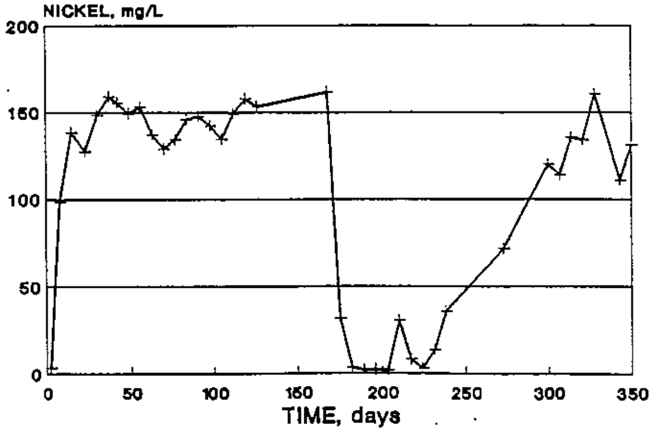

Figure 2. Average effluent nickel concentrations from two columns receiving aqueous solutions containing 200 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

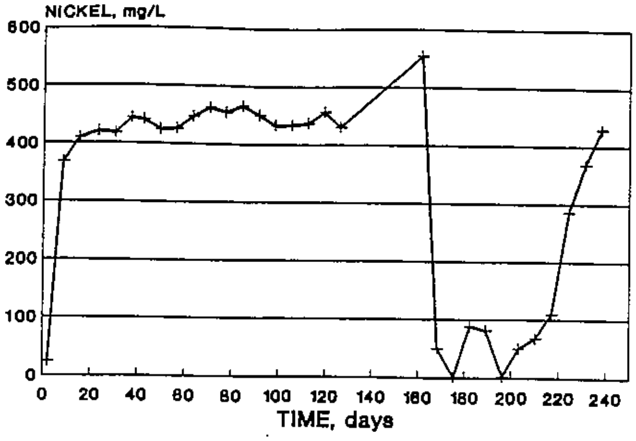

Figure 3. Average effluent nickel concentrations from two columns receiving aqueous solutions containing 500 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

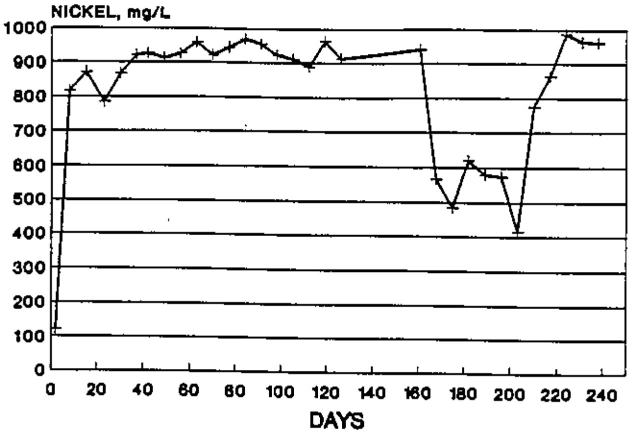

Figure 4. Average effluent nickel concentrations from two columns receiving aqueous solutions containing 1000 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

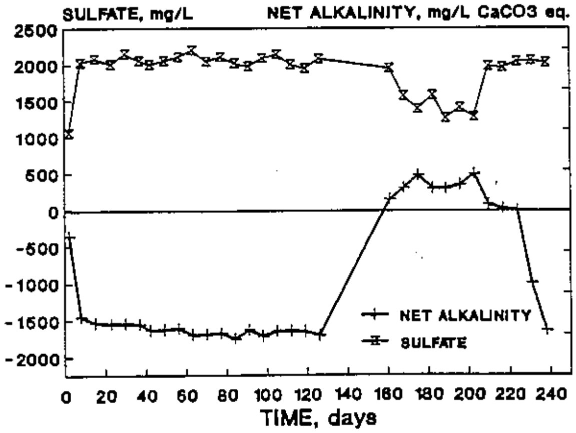

Figure 5. Average net alkalinity and sulfate concentrations in effluents from columns receiving solutions containing 100 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

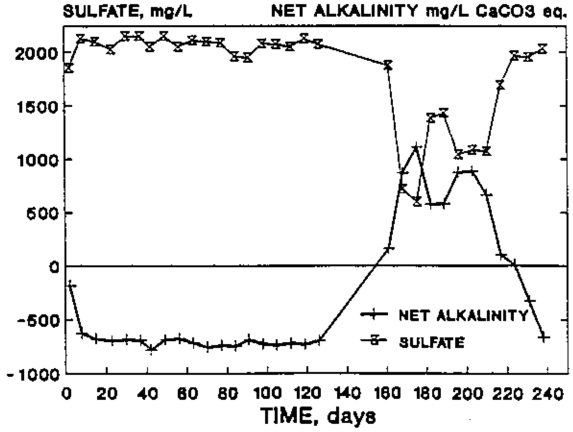

Figure 6. Average net alkalinity and sulfate concentrations in effluents from columns receiving solutions containing 200 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

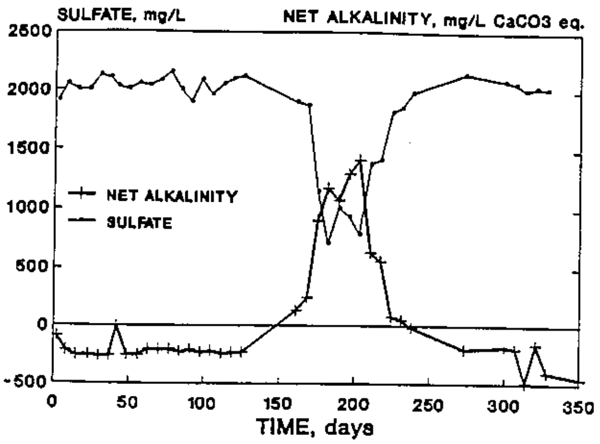

Figure 7. Average net alkalinity and sulfate concentrations in effluents from columns receiving solutions containing 500 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

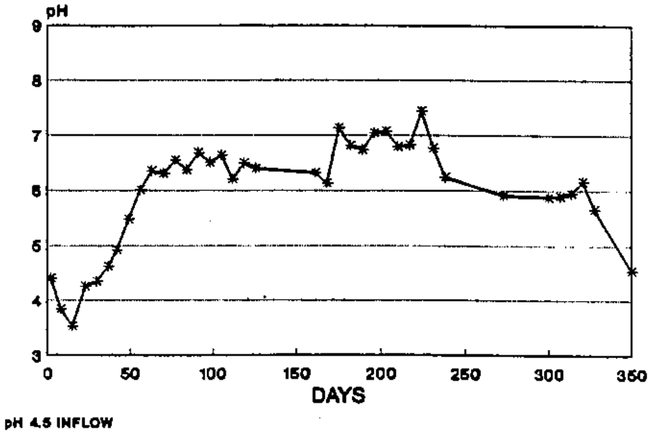

Figure 9. Effluent pH of a column receiving solutions containing 500 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

pH decrease, ion exchange can account for the removal of at least 5 mg Ni^{2+}/L, assuming that all cation exchange sites were occupied with H_3O^+ following acid washing of the compost. Nickel removal due to ion exchange may have been higher than indicated, considering that some of the released H_3O^+ may have been consumed subsequently by sulfate reduction.

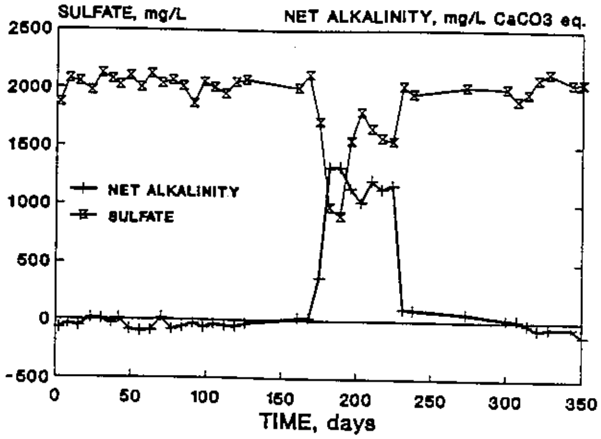

Figure 8. Average net alkalinity and sulfate concentrations in effluents from columns receiving solutions containing 1000 mg/L nickel and 2000 mg/L sulfate. Lactate supplementation (3500 mg/L) began on day 161 and was suspended on day 198.

Sulfate Reduction Phase

After day 9, the rates of nickel removal in the mushroom compost columns fell to 18-30 mg Ni/day (7.8-12.8 nmol/g-substrate/day) (fig. 1-5). These rates remained relatively stable until supplemental carbon was added on day 161. It is unlikely that sorption processes contributed to nickel removal during this time. Otherwise, the rate of nickel removal would have been expected to decrease with time and exhibit a greater dependence on the influent nickel content (fig. 10).

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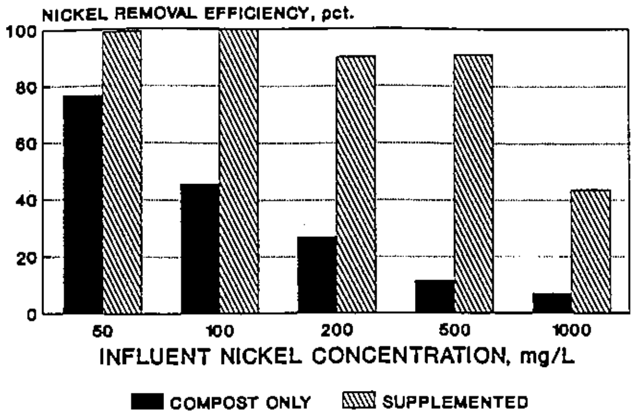

Figure 10. Average nickel removal rate (n = 14) at five influent nickel concentrations.

The increases in pH and alkalinity, the long-term and sustained rates of nickel removal, and the fact that nickel removal rates were only slightly dependent on influent nickel content all imply that sulfate reduction was probably responsible for the nickel removed during this period of time. However, no decrease in effluent sulfate concentration was detected. Nickel removal during this period ranged from 40 to 60 mg/L; about 65 to 98 mg/L of sulfate would have to be reduced to precipitate this amount of nickel as a sulfide. The dissolution of sulfate minerals (eg. gypsum) within the mushroom compost could easily have generated more sulfate than was consumed by these low rates of sulfate reduction. Therefore, it is not surprising that no net loss of sulfate was observed. There is no evidence to suggest that ion exchange processes contributed to nickel removal during this period.

Sulfate reduction rates in this study were estimated from measured rates of nickel removal although, during certain periods, nickel was removed from solution by sorption and ion exchange mechanisms, as well as sulfate reduction. However, nickel removal by mechanisms other than sulfate reduction quickly decreased as sorption and ion exchange sites become saturated. The nickel removal rates used for estimating sulfate reduction rates were determined after sorption and ion exchange mechanisms were no longer significant. These estimates assumed that all sulfide generated by sulfate reduction is precipitated as nickel sulfide. If hydrogen sulfide or bisulfide washed out of the columns without reacting with nickel, the estimated sulfate reduction rate could be significantly lower than the actual rate. Unfortunately, measurements of effluent sulfide concentrations were too infrequent to accurately determine the amount of unreacted sulfide that escaped from the columns. Therefore, estimated sulfate reduction rates are only a rough approximation of the actual rates, but do indicate the capacity of a compost system to precipitate metals. Sulfate reduction rates calculated from the amount of nickel removed were 10-20 times lower than rates measured previously in the mushroom compost substrate of a constructed wetland receiving acid mine drainage (McIntire et al. 1990). If nickel was inhibitory to sulfate-reducing bacteria, higher nickel concentrations would be expected to result in significantly lower sulfate reduction rates. However, sulfate reduction rates increased slightly with increased nickel content (fig. 10). This observation can be explained by the fact that higher metal contents result in lower concentrations of hydrogen sulfide or bisulfide which are known to be inhibitory to sulfate reducing bacteria (Miller 1972). Therefore, nickel toxicity to sulfate-reducing bacteria does not appear to be responsible for the low rates observed during this period. However, sulfate reduction rates may be limited by the ability of mushroom compost to supply labile carbon. The acid-washing of the mushroom compost during column preparation may have both removed labile carbon and destroyed fermentative bacterial populations necessary for the formation of labile carbon. To test this hypothesis, 11 mL of a 60 pct sodium lactate solution were added to each liter of inflow. Nickel removal and sulfate reduction rates increased seven-fold to a maximum of 216 mg Ni/day (92 nmol/g-substrate/day). Effluent sulfate concentrations dropped dramatically, and pH and alkalinity showed marked increases during the period of sodium lactate supplementation. The positive response of sulfate reduction indicators to the addition of sodium lactate supported the hypothesis that sulfate reduction on mushroom compost was carbon limited. The nickel removal efficiencies for columns operating with and without lactate supplementation are shown in figure 11.

The estimated sulfate reduction rates for the columns in the presence of excess labile carbon were lower than expected. However, because the estimated rates were based on nickel removal, the low rates are partly explained by the fact that some hydrogen sulfide or bisulfide exited the column without reacting with nickel. During the period of supplementation, sulfide concentrations as high as 196 mg/L were measured in column effluents. Increases in net alkalinity (fig. 5-8) corroborate the release of unreacted sulfide from the column because hydrogen sulfide must be removed from solution as a gas in order for alkalinity to increase. There would be no net increase in alkalinity when hydrogen sulfide or bisulfide was precipitated as nickel sulfide. In addition, anaerobic, purple sulfur bacteria were observed growing in the columns during the experiment. These photoautotrophic bacteria use light energy to convert hydrogen sulfide to elemental sulfur and hydrogen gas. The activity of purple sulfur bacteria would reduce the amount of hydrogen sulfide available for metal precipitation. For these reasons, all bacterially-generated sulfide would not be available for nickel precipitation. Therefore, actual sulfate reduction rates in the column may have been comparable to the 150-200 nmol/cc-substrate/day rates measured by McIntire et al. (1990).

Oxygen and pH inhibition of sulfate-reducing bacteria where water entered the mushroom compost columns may be indicated by lower AVS accumulations in these areas (fig. 12). Sulfate reduction cannot occur until all of the dissolved oxygen has been removed from the influent stream. At the influent pH of 4.5, sulfate reduction does occur but is probably greatly inhibited (Wakao

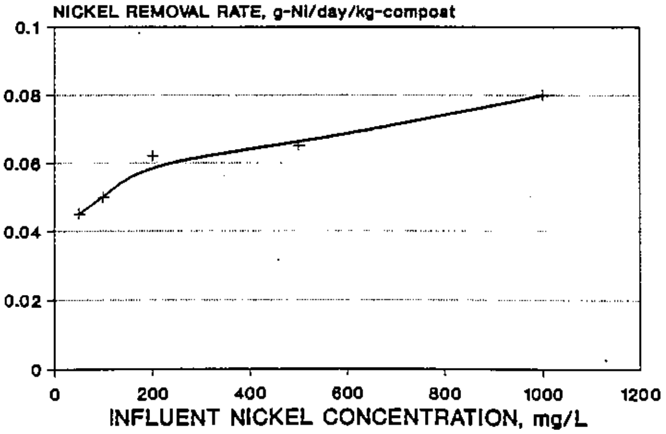

Figure 11. Nickel removal efficiencies for supplemented and unsupplemented columns at influent nickel concentration between 50 mg/L and 1000 mg/L.

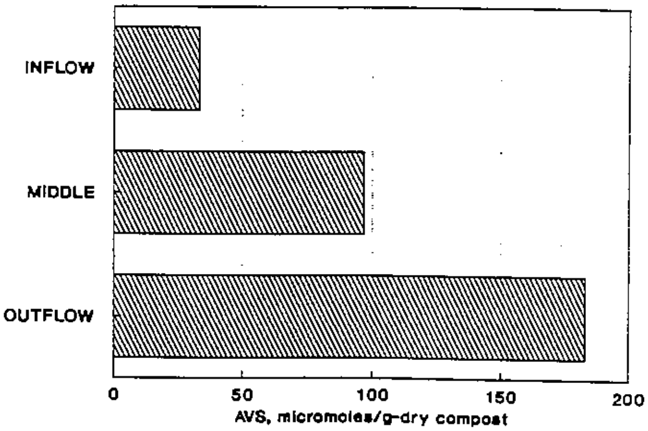

Figure 12. Average acid volatile sulfide accumulations in three segments of columns receiving solutions containing 500 mg/L nickel and 2000 mg/L sulfate.

1979). Results of this study (fig. 10 and 13) imply that nickel does not inhibit the activity of sulfate-reducing bacteria at concentrations at or below 1000 mg/L. Previous studies that evaluated the toxicity of nickel on sulfate-reducing bacteria have reported contradictory conclusions. Miller (1950) observed that nickel inhibited sulfate-reducing bacteria growing on sodium lactate. However, in a later study, Capone et al. (1983) found that the addition of 1000 mg/L nickel had no long-term effect on methanogenesis, sulfate reduction, and carbon dioxide evolution in anoxic salt marsh sediments. McIntire et al. (1990) reported no toxicity at nickel concentrations below 211 ppm. The extent to which nickel inhibits the activity of sulfate-reducing bacteria, if at all, cannot be concluded based on the results of the present study. However, at nickel concentrations less than 1000 mg/L, inhibitory effects appear to be minor and should not significantly impair sulfate reduction treatment methods.

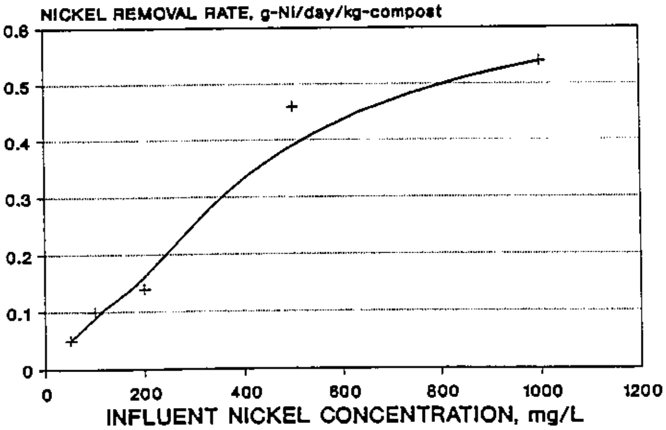

Figure 13. Average nickel removal rate (n = 6) at five influent nickel concentrations during lactate supplementation.

Conclusions

Sulfate reduction is a low-cost, low-maintenance technique capable of treating mine waters with elevated nickel concentrations. Based on 12-hr residence times, sulfate reduction treatment systems using mushroom compost as the primary organic substrate could remove 45-75 mg Ni^{+2}/kg-substrate/day from pH 4.5 wastewaters containing up to 1000 mg/L Ni^{+2}. Supplementation of the influent with 3500 mg/L lactate increased nickel removal to 540 mg Ni^{+2}/kg-substrate/day. Other less expensive industrial organic waste products may be useful in supplementing the nutrient requirements of sulfate-reducing bacteria in environments where their metabolic activity is sub-optimal. Peptone, glucose, and molasses (Wakao et al. 1979; Maree and Strydom 1987) are known to sustain sulfate reduction when fermentative bacteria are also present. Waste products of the food processing industry such as whey, brewery wort, sauerkraut filtrate, and peach peelings (Corrick et al. 1970) are low-cost supplements available in some areas. Anaerobically digested cattle wastes (Ueki et al. 1988) have widespread availability. Research is currently underway to identify other widely-available organic waste products that can be used by sulfate-reducing bacteria and their associated fermentative bacterial populations.

Two potential drawbacks to sulfate reduction-based treatment systems are that their efficiency will be decreased by cold temperature and low pH. Additional capacity must be engineered into systems to compensate for reduced biological activity in winter months. Placing systems underground where temperatures are more constant may result in more uniform performance, for example. Likewise, sulfate reduction treatment systems could be built large enough to provide enough inherent buffering capacity to compensate for short periods of time when the influent pH would normally be inhibitory to bacteria. These engineering design alternatives are currently being investigated (Dvorak et al. 1991).

Acknowledgements

The authors acknowledge the work of John W. Kleinhenz who performed the day-to-day maintenance of these experiments. We also thank Darryl H. Dvorak for AVS determinations on substrate samples.

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