# CONTROL OF ACID DRAINAGE FROM MINE WASTES

## USING BACTERIAL INHIBITORS<sup>1</sup>

## Bу

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Abstract. Detergents such as sodium lauryl sulfate (SLS) and food preservatives such as salts of benzoate and sorbate have been shown effective in controlling acid production from coal refuse. As part of the Bureau of Mines, U.S. Department of Interior's research of acid mine drainage, pilot-scale tests of these inhibitors was conducted on four types of mine waste: coal refuse, oxidized sulfide tailings, unoxidized sulfide tailings, and waste rock from a silver mine. In the experiment, two sample sizes were used, 7 kg and 100 kg. The 100-kg samples of each material were treated once with a 600-mg/kg dose of SLS or potassium benzoate. The 7-kg samples were treated with a high (600 mg/kg) or low (60 mg/kg) dose of SLS, potassium benzoate, or potassium sorbate. The treated samples and untreated control samples were subsequently leached once per week with filtered demineralized water (an amount equivalent to 2.54 cm of precipitation). The most successful inhibition was found in treatment of silver mine waste rock. For the 100-kg samples of the silver mine waste rock, SLS and potassium benzoate have completely inhibited iron-oxidizing bacteria such as Thiobacillus ferrooxidans for 28 and 29-plus weeks, respectively. (Testing of the silver mine waste rock is still in progress.) Cumulative acid loads from the SLS- and benzoate-treated samples of waste rock are 92 and 84 pct lower, respectively, than from the untreated control. Treatment of the 100-kg samples of coal refuse with SLS and benzoate showed short-term (1 to 7 week) bacterial inhibition. Cumulative acid loads remained 50 pct lower than the untreated control in the SLS- and benzoate-treated samples of coal refuse for 11 and 9 weeks, respectively. In the 100-kg samples, iron-oxidizing bacteria were inhibited by both treatments in the fresh sulfide tailings, but no inhibition was observed in the oxidized sulfide tailings. Neither treatment reduced leachate acidity levels in the 100-kg samples of oxidized and unoxidized sulfide tailings.

#### Introduction

The Bureau of Mines, U.S. Oepartment of Interior is currently researching at-source control of acid mine drainage (AMO). AMD is formed by water and oxygen reacting with sulfide minerals. Refuse and wastes from the mining of coal and metal ores contain many exposed pyrite surfaces, which eventually lead to the formation of AMD discharges. In the United States, approximately 3.5 million tons of acidic drainage

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is discharged per year, which results in contamination of surface and subsurface water supplies (Browning 1970).

A common remedy to AMO is chemical treatment of the contaminated water with alkaline materials such as lime, limestone, sodium hydroxide, or sodium carbonate. These treatments raise pH, reduce acidity, and precipitate metals. Such treatments are necessary until all of the available pyrite has reacted and the discharge water quality conforms to legal effluent limits. The length of time for a site to reach the point where all of the available pyrite has reacted is based upon many variables, but most estimates are measured in tens, hundreds or even thousands of years (Cathles 1982).

A more direct approach is to try to stop or slow the pyrite oxidation process. The generally accepted chemical reactions involved in the formation of AMD are as follows: ation, 1986 pp 123-130

123 https://doi.org/10.21000/JASMR86010123  $2FeS_2 + 70_2 + 2H_{20} - 2Fe^{2+} + 4S0_4^{2-} + 4H^+,(1)$ 

 $4Fe^{2+} + 0_2 + 4H^+ - 4Fe^{3+} + 2H_20$ , (2)

 $FeS_2 + 14Fe^{3+} + 8H_{20} - 15Fe^{2+} + 2SO_4^{2-} + 16H_{13}$ 

The sulfide moiety of pyrite (FeS2) is oxidized by oxygen (Eq. 1) resulting in ferrous iron (Fe<sup>2+</sup>), sulfate (SO4<sup>2-</sup>), and acid (H<sup>+</sup>). The ferrous iron can then be oxidized to ferric iron (Fe<sup>3+</sup>). The ferric iron can directly oxidize pyrite (Eq. 3), which results in more ferrous iron, sulfate, and acid. The rate-limiting step of abiotic oxidation is equation 2, which proceeds slowly in the absence of iron-oxidizing bacteria (Thiobacillus ferrooxidans) (Dugan 1975). Unfortunately, these bacteria, which are indigenous to areas where pyrite occurs, can accelerate the rate of equation 2 as much as a million times (Dugan 1975). In fact, the catalytic activity of these bacteria may be the cause of over 80 pct of the AMD problem in the United States (Browning 1970). Without the catalytic effect of these bacteria, water treatment costs can be reduced. Considering the resultant reduction in sludge handling and disposal costs, inhibiting the bacteria may become an even more cost effective alternative.

Various chemicals can be used to inhibit <u>I. ferrooxidans</u>. SLS and other surfactants have been shown to reduce AMD in laboratory-scale, pilot-scale and full-scale field tests on coal refuse (Stancel 1982, Kleinmann and Erickson 1982). One disadvantage of applying surfactants in solution form is the necessity to repeat the application every 3 to 6 months. To extend the duration of effective surfactant treatment, surfactants can be impregnated into rubber pellets for slow release of the chemical (Kleinmann and Erickson 1981). The success of these surfactant treatments, in solution or slow-release form, is dependent on many variables; most notably, the capacity of the refuse material to adsorb treatment, hydrologic characteristics, and severity of AMD.

In an attempt to identify chemicals that may have a longer effect when added as solutions, organic acids and their ability to form organic precipitates were studied (Onysko et al. 1984a). It was hypothesized that the organic precipitates formed would slowly redissolve and release small amounts of chemical inhibitor with each wetting. The idea was to use a natural mechanism for slow release similar to the artificial mechanism of surfactant-impregnated rubber pellets. A Bureau of Mines study found that sorbate and benzoate can be as effective as SLS on fresh coal refuse; however, no extension of AMD reduction was noted (Onysko et al. 1984b). The study also found that both sorbate and benzoate treatments are cost competitive with SLS treatment.

Because similar chemical reactions (Eqs. 1, 2, and 3) take place in metal mine wastes, inhibition of <u>T. ferrooxidans</u> should reduce acid production from these wastes. The intent of this Bureau of Mines, U.S. Department of the Interior study of acidic drainage formation in metal mine wastes was to determine (1) if these chemicals (detergents and organic acids) would inhibit the iron-oxidizing bacteria, (2) if inhibition of bacteria would reduce acid production, and (3) the effectiveness and duration of the chemical treatments at various dosages.

## Waste Materials

The waste materials tested were fresh coal refuse, oxidized and unoxidized sulfide tailings, and silver mine waste rock. All of these materials are common wastes resulting from coal and metal (copper, zinc, silver, and gold) mining. Coal refuse was included in the study since the effects of bacterial inhibition in coal refuse are well established.

The combined coal refuse (5 cm x 0 cm) used in this experiment was separated from the Freeport and Kittanning Coal Seams in southwestern Pennsylvania using standard coal processing devices: jigs, cyclones, heavy media and froth flotation. For the experiment, the refuse was collected immediately after discharge and transported to the Bureau test facility.

The unoxidized and oxidized sulfide tailings came from a metal mine in Quebec, Canada. The ore from this mine is passed through a concentrator and processed for recovery of copper, zinc, pyrite, gold, and silver. The ore passes through multiple crushers, grinders, and flotation cells. The waste product from the flotation cells constitutes the sulfide tailings (70 pct less than 200 mesh). The oxidized sulfide tailings used in the experiment were taken from the surface of a large tailings pile, and are easily identified as oxidized by their orange color. The unoxidized sulfide tailings, which were dark grayish brown, were taken from under the oxidized layer of the pile.

The silver mine waste rock came from a metal mine in British Columbia, Canada, where ore is mined for its silver, copper, and gold content. The material used in the experiment was unprocessed since it was below ore grade. The material was unsized (15 cm x 0 cm) and represented the most acid-producing material from this mine.

Samples of each material were taken at the beginning of the experiment for sulfur determination. Total sulfur was measured using the combustion furnace method. To differentiate the various sulfur forms, two acid extractions, one with hydrochloric acid and one with nitric acid, were performed. The residues from these extractions were analyzed in a combustion furnace, and the percentages of total, pyritic, sulfate, and organic sulfur were calculated (table 1).

#### Experimental Procedures

The criteria for the design of the experiment were (1) to have backup samples to show repeatability of results, (2) to test three inhibitors, and (3) to test two treatment doses. The supply of waste materials limited the experiment to three 100-kg samples of each waste material: one untreated control, one treated with SLS, and one treated with potassium benzoate. In addition to these 100-kg samples, eight 7-kg samples of each material were treated as follows: two untreated controls, a high and low dose of SLS, a high and low dose of potassium benzoate, and a high and low

Material	Sulfur form, pct					
	Total	Pyritic	Sulfate	Organic		
Coal refuse	8.65	7.70	0.19	0.76		
Unoxidized sulfide tailings	20.50	19.73	0.84	-		
Oxidized sulfide tailings	5.98	0.17	5.81	-		
Silver mine waste rock	4.07	4.07	-	-		

Table 1. Sulfur Content of Waste Materials

dose of potassium sorbate. The experimental design is summarized in table 2.

For the 100-kg samples, fifteen 200-L plastic barrels were set up on wooden platforms. Two holes were drilled in the bottom of each barrel, and a plastic bucket was placed underneath each hole to collect leachate. For the 7-kg samples, forty 20-L plastic buckets were used. Each bucket had two holes drilled in the bottom and was positioned over another bucket to enable leachate collection.

Each of the four mine wastes was used to fill three barrels and eight buckets with 100 kg and 7 kg of material, respectively. The waste materials were slowly added (without compaction) to their respective containers, and within 1 week, the experiment began. An enclosed hangar-type building in which the air temperature ranged from approximately 15 to 25° C was used for the experiment.

Each barrel (100-kg samples) designated to receive chemical treatment was treated with 8 L of a 7,500-mg/L solution of SLS or benzoate. These treatments were equivalent to 600 mg of treatment chemical per kilogram of waste material. The barrels designated as controls received 8 L of deionized tap water.

Two of the eight buckets (7-kg samples) of each material were designated as untreated controls. The remaining six buckets of each material received a high or low dose of SLS, potassium benzoate, or potassium sorbate. The high treatment dose was added to each bucket using 1.2 L of the appropriate 3,500-mg/L solution. The low treatment dose was applied using 1.2 L of a 350-mg/L solution. The high and low treatment doses were equivalent to 600 and 60 mg, respectively, of treatment chemical per kilogram of material. The buckets designated as controls received 1.2 L of deionized tap water.

Treatment dose has been given in weight of chemical treatment per weight of waste material treated. It is common to express field doses in gallons of a 30-pct solution of treatment chemical per acre of surface treated. Based on the surface area of the samples, the 600-mg/kg treatment dose for the 100-kg samples was equivalent to 730 gal of 30-pct treatment solution per acre. For the 7-kg samples, the high and low treatment doses (600 mg/kg and 60 mg/kg) were equivalent to 290 and 29 gal of 30-pct chemical solution per acre.

The weekly leaching program began 1 week after treatment. Tap water was passed through a 0.20- m capsule filter and a two-bed ion-exchange-resin system before storage in a 500-gal plastic reservoir. Periodic sampling of this reservoir showed the water to be acceptably low in dissolved solids, and the population of iron-oxidizing bacteria was below detectable limits (<1 cell/100 mL). A plastic sprinkling can was used to evenly distribute water on each waste material. The 100-kg and 7-kg samples were leached with 8.0 and 1.2 L of water, respectively; these volumes are equivalent to 2.54 cm of

Sample weight,	Chemical	Treatment	Volume of water used
kg	treatment	dosage, mg/kg	in weekly leachings, L
	I T		
100	Untreated.	-	8.0
$100\ldots$	SLS	600	8.0
100	Benzoate	600	8.0
7	Untreated.	-	1.2
7	1do		1.2
7	SLS	600	1 1.2
7	do	60	i 1.2
7	Benzoate	600	1.2
7	do	60	1.2
7	Sorbate	600	1.2
7	do	60	1.2
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Table 2. - Treatment of Samples for Each Waste Material

Material	Treatment	Duration of bacterial inhibition for 100-kg	Duration of Acidity Reduction <sup>B</sup> , we be a second se		
	chemical	samples <sup>A</sup> , weeks	100-kg samples	High dose	Low dose
	SLS	7	6	8	0
Coal refuse l	Benzoate	1	5	5	0
	Sorbate	-	-	5	
Unoxidized	SLS	22	0	0	0
Sulfide	Benzoate	18	0	1 0	1 0
Tailings	Sorbate	-	0	0	0
 Oxidized	SLS	0	0	0	i o
Sulfide	Benzoate	Ō	0	0	0
Tailings	Sorbate	-	0	0	0
Silver mine.	SLS	28	29+	29+	1 8
Waste rock	Benzoate	29+	29+	20	12
	Sorbate	_	-	20 	

# Table 3. - Summary of Results

ALength of time bacteria were inhibited to less than 1 pct of the control.

 $B_{\mbox{Length}}$  of time leachate acidity was less than 50 pct of the control.

#### precipitation on the waste material surface.

After the waste materials were watered, they freely drained into the sample- collection buckets for 24 h. Samples from each leachate were taken and the remaining volume was measured and discarded. To reduce labroatory analyses, samples from alternate weeks were refrigerated for 7 days and combined with an equal volume of a corresponding sample from the subsequent leaching. These composite samples were then analyzed.

Water samples of the leachate from each bucket and barrel were analyzed for pH, acidity, ferrous iron, total iron, calcium, magnesium, aluminum, sodium, mangamese, and sulfate. Iron was detected with a potassium dichromate titration using the sodium salt of diphenylaminesulfonic acid as an indicator. Other metal concentrations were determined by inductively coupled argon plasma (ICAP) spectroscopy. After passing the sample through a cation-exchange resin, sulfate was analyzed by barium chloride titration using thorin as an indicator. Acidity analysis consisted of a fixed-end-point titration to pH 8.3 using a 1N sodium hydroxide solution. Iron-oxidizing bacterial populations were estimated for the barrels only. The multipletube, most probable number (MPN) technique described by Olem and Unz (1977) was used.

## Results

The following sections cover the results of the three treatment chemicals on the four mine waste materials. Although direct measurements of the bacterial populations were made, the effectiveness of the chemicals is based on leachate water quality. Graphs of acidity concentration and cumulative acid load over time are presented since acidity directly relates to water treatment costs. Sulfate and total iron consistently followed the same trends as acidity. The results are summarized in table 3.





## Coal Refuse

All three chemicals - SLS, benzoate, and sorbate - reduced acidity levels in the coal refuse when applied at the 600-mg/kg dose. Cumulative acid loads from the SLS-treated 100-kg sample remained less than 50 pct of the acid load from the control for 11 weeks (fig. 1). The ironoxidizing bacteria were totally inhibited by the SLS for 8 weeks. After the 8-week period, the bacteria repopulated very guickly to levels equal to that of the control, where the iron-oxidizing bacterial population ranged from 1 to 5 million cells per 100 mL of leachate. SLS treatment in the 7-kg samples showed that the high dose (600 mg/kg) reduced acidity for approximately 8 weeks, but the low dose (60 mg/kg) had no observable effect (fig. 2).

Potassium benzoate also reduced acidity levels in the 100-kg sample. Cumulative acid load from the benzoate-treated samples remained below 50 pct of the untreated control for 9 weeks (fig. 1). The bacteria were only inhibited for 1 week, with a subsequent increase in population to levels found in the control. Results from the 7-kg













samples showed the high dose of benzoate reduced acidity for 5 weeks, and the low benzoate dose had no apparent effect (fig. 2).

Only the 7-kg samples received potassium sorbate treatment. The high treatment dose of sorbate reduced acidity levels for about 5 weeks. The effectiveness of the high dose of sorbate was similar to that of the high dose of benzoate and SLS in terms of acidity reductions. The low dose of sorbate had no effect.

# Unoxidized Sulfide Tailings

None of the three chemicals effectively reduced acidity levels in the leachate from the unoxidized sulfide tailings. In fact, acidity levels were higher from the SLS-treated sample than from the untreated control, despite signifi-cant bacterial inhibition. The initial acidity concentration for the SLS-treated sample was more than four times that of the control (fig. 3). Acidity levels remained higher than that of the control for 10 weeks, and approximately equaled the control for the remainder of the experiment. The SLS totally inhibited the iron-oxidizing bacteria for 8 weeks and continued to keep the bacterial population below that of the control for an additional 14 weeks. The population of iron-oxidizing bacteria in the untreated control ranged from 3.5 to 30 million cells per 100 mL. Neither high nor low doses of SLS on the 7-kg samples had any significant effect on leachate quality (fig. 4).

Potassium benzoate treatment also increased acidity levels in the leachate from the 100-kg sample while keeping the bacterial population below that of the control for 18 weeks. Over a threefold increase in acidity levels were found in the leachate from the benzoate treatment compared with that of the control (fig. 3). Neither high nor low doses of benzoate or sorbate affected leachate acidity levels of the 7-kg samples.

# Oxidized Sulfide Tailings

Results from the oxidized sulfide tailings were similar to the results from the unoxidized tailings. In 100-kg samples, greater concentrations of acidity were leached from the SLS- and benzoate-treated samples than from the control in the first 6 to 8 weeks (fig. 5). But unlike the unoxidized material, bacterial populations were



not inhibited, and ranged from 3,000 to 230,000 cells per 100 mL. Results from the 7-kg samples show that all of the treatments increased acidity levels in the first 4 to 8 weeks of the experiment with no subsequent difference in acidity levels between treated samples and controls for the remainder of the experiment.

#### Silver Mine Waste Rock

Both high and low doses of all treatments reduced acidity levels in the silver mine waste rock. Results are reported through week 29, with the experiment still in progress. After 29 weeks, cumulative acid loads from the SLS-treated and untreated control 100-kg samples were 103 g and 1130 g, respectively (fig. 6). This represents a 92-pct reduction in cumulative acid load for the SLS treatment (fig. 7). Iron-oxidizing bacteria were totally inhibited for 28 weeks, with repopulation beginning in week 29. Bacterial population in the control ranged from 25,000 to 300,000 cells per 100 mL during the first 29 weeks. The low treatment dose of SLS on the 7-kg samples kept weekly acidity less than 60 pct of control levels for 15 weeks; the high dose continues (after 29 weeks) to keep acidity levels significantly (>98 pct) below those of the controls (fig. 8).

Benzoate treatment of the 100-kg samples has reduced cumulative acid loads 84 pct over that of the control after 29 weeks. Although a greater reduction of cumulative acidity was found from the SLS treatment during the first 29 weeks, the most recent data show that acidities from the benzoate treatment continue to decrease while acidities from the SLS treatment are increasing (fig. 6). In the first 2 weeks of the experiment, benzoate treatment did not totally inhibit the bacteria, and relatively high acidity levels were observed. From week 3 to 29, the bacteria were totally inhibited by the benzoate treatment. Benzoate treatment of the 7-kg samples reduced weekly acidity concentrations to less than 60 pct of the level of the controls for 13 and 22 weeks for the low and high treatment doses, respectively (fig. 8).

The low dose of potassium sorbate did not reduce acidity in the 7-kg samples. As a result of the high sorbate dose, acidity was reduced to less than 60 pct of the levels in the controls for 22 weeks (fig. 8).

Results from the 100-kg samples show that the benzoate treatment is lasting longer than is the SLS treatment, but the high dose of SLS is lasting longer than the high dose of benzoate in the 7-kg samples. Although the high treatment dose used in the 7-kg samples was the same as the treatment dose in the 100-kg samples (600 mg/kg), the concentrations used to add these treatments differed: 3,500 mg/L for the 7-kg samples and 7,500 mg/L for the 100-kg samples.

## Discussion

Variations of acidity concentrations over time for the untreated controls of the 100-kg samples are shown on figure 9. Material processing, sulfur content, extent of weathering, and size distribution are the major characteristics that determine the various shapes for these



Figure 6. Silver mine waste rock, 100-kg samples.











Figure 8b. Silver mine waste rock, 7-kg samples, low treatment dose (60 mg/kg).



Figure 9. Untreated controls, 100-kg samples.

graphs. The leaching procedure used in this experiment represents an acceleration of actual field conditions. Although 2.54 cm of precipitation does not seem excessive, the procedure does not allow for runoff or significant evaporation that might occur in the field. The duration of bacterial inhibition for the treatment chemicals is also affected by the short period of time the water is added. Although this may increase the amount of chemical that is washed off, the treatment is not lost to runoff and may be deposited at a lower position in the sample. In any case, caution must be exercised when comparing the results of laboratory leaching tests to intended field use. Simple adsorption tests can also provide information concerning the suitability of the various chemical inhibitors on particular waste materials (Kleinmann and Erickson 1982).

Results from the coal refuse show that all three chemicals applied at the 600 mg/kg dose kept the concentration of acidity below 50 pct of the untreated control for 5 to 8 weeks, but the actual rate of acid production (shown by the slope of the graphs in figure 1) was delayed only 4 weeks. In additional adsorption tests, this material was found to have relatively low SLSadsorption capacity: two to eight times less than that of other coal refuse tested.

Acidity was not reduced in any of the treated samples of unoxidized sulfide tailings, but the iron-oxidizing bacteria were inhibited by both SLS and benzoate in the 100-kg samples for about 20 weeks. Acidity concentrations were unaffected by the treatments in the 7-kg samples, but were increased by the treatments in the 100-kg samples. This discrepancy can be explained by the drainage characteristics of the samples. All of the 7-kg samples drained uniformly. Of the 100-kg samples, however, the control drained much faster than did the SLS- and benzoate-treated samples, resulting in lower acidity values. The results of the 7-kg samples of unoxidized sulfide tailings showed that these bacterial inhibitors did not significantly reduce or increase contaminant levels.

Results from the oxidized sulfide tailings show that all of the treatments increased acidity levels in 100- and 7-kg samples. The material is considerably oxidized with 5.81 pct sulfate sulfur and only 0.17 pct pyritic sulfur; therefore, most of the contamination in the leachate is from the flushing of previously oxidized material. Apparently, the treatment chemicals caused an increased flushing of contaminants. Therefore, none of these bacterial inhibitors are applicable for field use on oxidized sulfide tailings.

Results from the benzoate- and SLS-treatments on the silver mine waste rock indicate that both chemicals may be applicable for field use. SLS has been applied by the mining company in the field (not as a part of this study), but no reductions in contaminant levels were observed. Their field dose was 45 gal of 30-pct SLS solution per acre. This field treatment dose was 16 times less than that used on the 100-kg samples, 7 times less than the high dose of the 7-kg samples, and 1.5 times greater than the low dose of the 7-kg samples. The blocky nature of the silver mine waste rock probably permits air to permeate deeply into the pile. Consequently, in order to inhibit bacteria and reduce contaminant levels, a large portion of the pile must receive the chemical inhibitor and not just the uppermost layer.

The capacity for a waste material to adsorb the bacterial inhibitor directly affects the duration of bacterial inhibition. Simple laboratory tests to determine the adsorption capacity of a waste material (Kleinmann and Erickson 1982) can provide valuable information concerning the probability of successful field application. Reductions in bacterial population and acidity concentration have significance only if the chemical inhibitor persists long enough to make the application of these chemicals cost effective.

#### Summary

SLS, potassium benzoate, and potassium sorbate effectively reduced acidity levels in the leachate from the silver mine waste rock. Acidity was reduced by the 600-mg/kg treatment dose for 20 to 29-plus weeks. (Duration of acidity reduction is based on length of time acidity concentrations from the treated samples reamined less than 50 pct of the untreated control.) The 60-mg/kg dose of sorbate did not reduce acidity. The 60-mg/kg dose of SLS and benzoate reduced acidity for 8 and 12 weeks, respectively. In the 100-kg samples, the ironoxidizing bacteria were totally inhibited for 28 and 29-plus weeks for the SLS- and benzoatetreated samples, respectively.

The three treatment chemicals also reduced contaminant levels in the leachate from the coal refuse. The 600 mg/kg treatment dose reduced acidity for 5 to 8 weeks. None of the inhibitors were effective at the 60-mg/kg dosage level. For benzoate and SLS treatment of the 100-kg samples, iron-oxidizing bacteria were inhibited for 1 and 8 weeks, respectively.

The treatments did not reduce contaminant levels in the leachate from the unoxidized sulfide tailings or oxidized sulfide tailings. The treatment chemicals increased acidity levels in the leachate from oxidized sulfide tailings for the first few weeks. The iron-oxidizing bacteria were inhibited for approximately 20 weeks by SLS and benzoate in the 100-kg samples of unoxidized sulfide tailings, but they were not inhibited in the oxidized sulfide tailings.

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