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REMOVAL OF HEAVY METALS FROM MISSOURI LEAD MILL TAILINGS BY FROTH FLOTATION

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ABSTRACT

The U.S. Bureau of Mines investigated froth flotation techniques to remove heavy metals (Pb, Cu, and Zn) from southeast Missouri lead mill tailings. It has been estimated that southeast Missouri contains between 200 and 300 million st of Pb tailings stored above ground. The tailings were classified as two distinct types: (1) pre-1968 tailings from the Old Lead Belt (some more than 100 years old) and (2) post-1968 tailings from the New Lead Belt. The objectives of the investigation were to reduce the Pb remaining in the tailings to <500 ppm (<0.05 pct Pb) and to attempt to recover a marketable concentrate to offset a portion of the remediation costs. The remaining dolomite-limestone would then be used as mining backfill or agricultural limestone. Bench-scale froth flotation removed, in percent, 95 Pb, 84 Cu, and 54 Zn, leaving 94 pct of the original weight containing, in parts per million, 400 Pb, 40 Cu, and 300 Zn from the Old Lead Belt tailings. Separate flotation tests also removed, in percent, 85 Pb, 84 Cu, and 80 Zn, leaving 75 pct of the original weight containing, in parts per million, 400 Pb, 200 Cu, and 500 Zn from the New Lead Belt tailings. Concentrates recovered from the Old Lead Belt were retreated to produce a final Pb concentrate containing 72 pct Pb with a cleaner flotation recovery of 79 pct. Froth flotation proved to be a viable method to remove the heavy metals.

INTRODUCTION

The Environmental Technology Division of the Bureau investigated froth flotation techniques for the removal of heavy metal values from southeast Missouri lead mill tailings. The tailings are the waste material from the processing of Pb

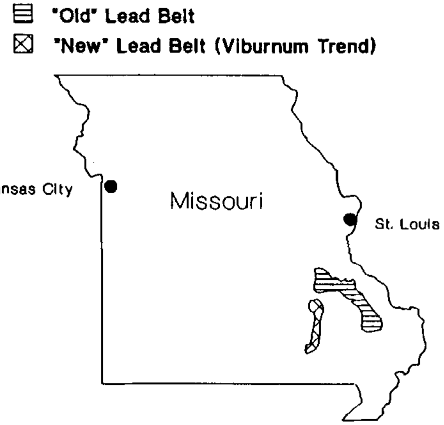

FIGURE 1. Location of Missouri's "Old" and "New" Lead Belts.

ores. Tonnage estimates for the tailings range from 200 to 300 million st. The southeast Missouri lead mill tailings can be divided into two groups: (1) the pre-1968 tailings, which were generated from the Old Lead Belt situated in Washington, St. Francois, and Madison counties, and (2) the post-1968 tailings, which are currently being generated from the New Lead Belt in southern Washington, Iron, and Reynolds counties. The New Lead Belt generates approximately 9 million st of new fresh tailings per year.

The older tailings are stored above ground in piles and pose a potential health and environmental problem due to their heavy metal content. This environmental problem is compounded by their proximity to populated areas and waterways. Due to the small size of a portion of the tailings (some <50 μm), they are prone to become airborne during windy conditions or eroded into waterways during periods of heavy precipitation. The Pb mill tailings from the New Lead Belt are contained at the mine site in tailings ponds or lagoons.

As an illustration to the age of some of the tailings, a brief history of Pb mining in southeast Missouri is needed. A generalized map of Missouri, showing the Old and New Lead Belts, is presented in Figure 1. In 1720, the deposit at Mine La Motte was discovered and production started immediately. The French continued exploration, and this led to the discovery of other Pb deposits in Washington County, near Potosi (*1*). In 1864, the Pb deposit at Bonne Terre was being mined, and by 1880, Pb was being mined at Desloge (*2*). Late in the 18th Century, Pb was discovered in St. Francois County near the Big River. These discoveries were followed by discoveries near the cities of Flat River and Farmington. Lead continued to be mined from the Old Lead Belt until the 1960's. In 1955, near Viburnum, new Pb deposits were discovered and production started in 1960 (*1*). These series of deposits would become known as the Viburnum Trend. By 1968, Pb was being mined along the whole length of the trend in what would become the New Lead Belt. Production of Pb in Missouri has continued almost uninterrupted for over 250 years. This investigation was conducted with the objectives of: (1) lowering the Pb remaining in the reprocessed tailings to <500 ppm (0.05 pct) and (2) recovering a marketable product to offset a portion of the potential remediation costs. The first objective of <500 ppm Pb remaining in the reprocessed tailings is in accordance with U.S. Environmental Protection Agency directives for Pb remaining in residential soils. In keeping with these objectives, the weight percent reporting to the reprocessed tailings product would need to be as high as possible and contain <500 ppm Pb, in order to maximize the grade of the concentrate for further cleaning. Therefore, the reprocessed tailings would virtually be the final product, and if a marketable product could be recovered, it would only enhance the economic aspect of the process. The reprocessed tailings might then be used as mining backfill for ground stabilization or as agricultural limestone.

OLD LEAD BELT TAILINGS

National Tailings

The National Lead Company mined and processed Pb ores in the Flat River, MO, area around the turn of this century (*3*). The processing methods used at that time included jigs and shaking tables. These methods were somewhat inefficient, thus their tailings were a coarse product containing small amounts of heavy metal values. The tailings were stockpiled at the processing site. In the following years, the town grew around the tailings pile.

The jig tailings pile at the National site encompasses 45 acres and is approximately 1,900 ft long, 1,050 ft wide, and 180 ft high. The pile is elliptical in shape with the long axis running east to west and has a dome on the eastern half. About 16 acres of slime pits are located on the northeast corner and the eastern side. The entire site encompasses approximately 321 acres, including the area developed by industry (4). Tonnage estimates for the National pile range from 6.5 to 10 million st. Samples of the tailings were taken using a wheel-mounted, gasoline-powered auguring device which allowed sample retrieval to a depth of 21 ft. Chemical analyses of the tailings show large variations in the metal value content from sample to sample. From the samples taken by the Bureau, the National pile contains on the average, in percent, 0.87 Pb, 0.035 Cu, 4.6 Fe, 0.11 Zn, 0.029 Co, and 0.029 Ni. Mineralogical examination revealed the sulfide minerals galena, chalcopyrite, sphalerite, marcasite, cobaltiferous pyrite, and nickeliferous pyrite, with the gangue being massive dolomite and a small amount of limestone. Almost all of the sulfides have an oxidation layer or show partial alteration. In the case of galena, it has been estimated that between 15 and 20 pct has altered to anglesite ($PbSO_4$) or cerussite ($PbCO_3$). The liberation size for the sulfides from the gangue was determined to be 100 mesh (150 µm). Table 1 shows a typical screen analysis of the National tailings.

Size analyses of the pile show large variations for the average particle size. These variations range from 950 to 6,250 µm. The large particle size would coincide with tailings from a gravity separation plant. In the coarser samples, >75 pct of the Pb values are present in the plus 12-mesh fraction (+1,700 µm).

TEST PROCEDURES

Feed Preparation

Laboratory-scale equipment was used in all the testing procedures. Initial testing involved milling to liberation with a rod mill. The length of the rod mill was 8.75 in and the outside diameter was 8.5 in. The mill was charged with six 0.5-in-

TABLE 1. SCREEN ANALYSIS OF NATIONAL TAILINGS

Mesh	Size, µm	Wt Pct	Pct Pass	Chemical Analyses, pct					
				Co	Cu	Fe	Ni	Pb	Zn
Plus 4.............	+4,750	27.31	72.69	0.01	T	4.5	0.02	0.34	0.04
Minus 4 plus 8......	+2,360	42.86	29.83	.01	T	4.6	.02	1.10	.06
Minus 8 plus 12.....	+1,700	8.58	21.25	.01	0.02	4.6	.02	1.20	.09
Minus 12 plus 16....	+1,180	4.54	16.71	.01	T	4.7	.02	.80	.05
Minus 16 plus 30....	+600	4.47	12.24	.01	.07	4.7	.02	.44	.06
Minus 30 plus 40....	+425	1.49	10.75	.01	.04	4.6	.02	.32	.07
Minus 40 plus 50....	+300	1.53	9.23	.01	.04	4.5	.02	.30	.10
Minus 50 plus 70....	+212	1.24	7.99	.01	.06	4.6	.01	.34	.09
Minus 70 plus 100...	+150	1.19	6.80	.01	.05	4.4	.01	.24	.09
Minus 100 plus 150..	+106	1.24	5.56	T	.07	4.4	T	.36	.11
Minus 150 plus 200..	+74	.77	4.79	T	.07	4.5	T	.36	.11
Minus 200..........	-74	4.79	0	.04	.30	4.1	.04	1.50	.25
Totals...........	NAp	100.00	NAp	0.011	0.026	4.5	0.02	0.82	0.07
				Distribution, pct					
Plus 4.............	+4,750	NAp	NAp	24.13	4.18	27.01	26.78	11.34	16.01
Minus 4 plus 8......	+2,360	NAp	NAp	37.88	6.56	43.33	42.04	57.59	37.68
Minus 8 plus 12.....	+1,700	NAp	NAp	7.58	6.56	8.67	8.41	12.58	11.31
Minus 12 plus 16....	+1,180	NAp	NAp	4.01	.69	4.69	4.45	4.44	3.33
Minus 16 plus 30....	+600	NAp	NAp	3.95	11.95	4.61	4.38	2.40	3.93
Minus 30 plus 40....	+425	NAp	NAp	1.32	2.28	1.50	1.46	.58	1.53
Minus 40 plus 50....	+300	NAp	NAp	1.35	2.33	1.51	1.50	.56	2.24
Minus 50 plus 70....	+212	NAp	NAp	1.09	2.84	1.25	.61	.51	1.63
Minus 70 plus 100...	+150	NAp	NAp	1.05	2.28	1.15	.58	.35	1.57
Minus 100 plus 150..	+106	NAp	NAp	.44	3.32	1.20	.24	.54	2.00
Minus 150 plus 200..	+74	NAp	NAp	.27	2.05	.76	.15	.34	1.24
Minus 200..........	-74	NAp	Nap	16.93	54.95	4.31	9.39	8.77	17.54
Totals...........	NAp	NAp	NAp	100	100	100	100	100	100

NAp Not applicable.
T Trace (0.004 pct for calculation).

diam rods, seven 0.625-in-diam rods, and eight 1-in-diam rods. The milling procedure consisted of a two-stage grind of the as-received tailings with the undersize (minus 100 mesh) removed after the first stage. This would ensure fresh mineral surfaces for subsequent flotation testing. The feed for each test was 500 g and was milled at 50 wt pct solids. The oversize (plus 100 mesh) remaining after the first stage was approximately 40 to 50 pct of the original weight and was reground at 50 wt pct solids. The two-stage milling procedure reduced the amount of fines generated. The normal grinding times were 10 min for the first stage and 5 min for the second stage.

Rougher Froth Flotation

The standard flotation procedure consisted of pulping the feed to 20 pct solids, addition of appropriate additives, pH adjustment (if needed), addition of collector, addition of frother, and introduction of air. The normal conditioning time was 3 min per addition.

Initial flotation tests were performed to screen various collectors. In these tests, attempts were made at recovering a bulk sulfide concentrate. One of the first situations noticed was a marked increase in Pb recovery compared to tests performed on the New Lead Belt tailings. Part of this may have been due to the fact that more Pb values were present in the older tailings or that the first collectors used on the older tailings were specifically for oxidized or tarnished minerals (5). Recoveries of the Pb values were in the range of 80 to 85 pct with Cu and Zn recoveries in the range of 60 to 80 pct. However, these tests did not lower the Pb content remaining in the reprocessed tailings to below the target value of 500 ppm. Emphasis was shifted to selectively recover the Pb values in order to meet the objectives of the study.

With this in mind, more selective collectors were investigated. One such collector system was a combination of thiocarbanalid and a xanthogen formate. When this collector combination was added to the grinding circuit with sodium sulfide, creosote oil, and a frother, the Pb recovery increased to 90 pct. The Cu recovery was 78 pct and the Zn recovery dropped to 21 pct; however, the reprocessed tailings still contained >800 ppm Pb.

These results were encouraging and pointed to the need for a multiple collector reagent scheme. Operating under this scenario, various collector combinations were tested in multiple-stage flotation tests. Various additives were introduced ahead of the collectors.

The best results were obtained using four flotation stages with two collectors and one collector mixture. The first two flotation stages employed sodium sulfide for activation (0.59 lb/st), a mixture of mercapthobenzothiazole and a dithiophosphate as the collector (0.23 lb/st), and MIBC (methyl isobutyl carbinol) as the frother (0.39 lb/st). The flotation pulp pH was 8.5 with these additions, and the total flotation time for the first two stages was 8 min. The next two flotation stages employed Cu sulfate for activation (0.98 lb/st), sodium diisopropyl dithiophosphate as one collector (0.23 lb/st), and sodium isobutyl xanthate as the second collector (0.23 lb/st). A frother was not needed in either of the last two flotation stages due to carry-over from the first two stages. For the last two stages, the flotation pulp pH was maintained at 10.0 with the addition of sodium hydroxide, and the total flotation time was 12 min.

This flotation system recovered 95 pct of the Pb values, 84 pct of the Cu values, and 54 pct of the Zn values. The final reprocessed tailings contained 94 pct of the original weight and were left with only 400 ppm Pb, 40 ppm Cu, and 300 ppm Zn remaining in the tailings. The combined concentrate grade was, in percent, 13.61 Pb, 0.36 Cu, 0.65 Zn, and 4.05 Fe. The high Pb grade of the combined concentrate represented an enrichment ratio of over 15.0. Since the grade of the combined concentrate was >5 pct Pb (typical run of mine ore is 5 to 10 pct Pb), the prospect of being able to upgrade the concentrate even further to recover a salable Pb concentrate looked good. This reagent scheme proved that the target value of <500 ppm Pb remaining in the reprocessed tailings could be met and even surpassed.

Continuous Bench-Scale Rougher Flotation

Using the four-stage flotation scheme just described, a bench-scale continuous rougher flotation circuit was constructed. The purpose of the circuit was to recover enough concentrate to perform cleaner flotation tests in order to attempt to enrich the Pb grade to a salable product. Due to space and equipment limitations, it was decided to use the reagent scheme but use only two flotation stages. As previously described, approximately 18 kg of tailings were stage milled in a laboratory rod mill. The flotation circuit consisted of a 100-L conditioning tank, two laboratory flotation cells, two 5-L conditioning tanks, and various small slurry pumps. The solids flowrate through the circuit was 175 g/min.

The reagent dosages for the continuous test were changed to attempt a more specific separation (mainly recover less Zn values) for the subsequent cleaning tests. With less Zn values present in the feed to the cleaner tests, the final Pb cleaner concentrate would not be diluted by those same Zn values. Since with an excess of sodium sulfide less Zn value tends to be recovered, the amount of sodium sulfide was increased by a factor of 6. As insurance for less Zn recovery, zinc sulfate was added, as a depressant, at a dosage of 0.1 lb/st. The collector dosages were also halved to determine if less reagent could be used. The continuous circuit recovered 85.5 pct of the Pb and 84.5 pct of the Cu at a combined Pb grade of 6.8 pct Pb. These results were acceptable for further cleaner flotation test feed.

Cleaner Flotation

Cleaner flotation testing was performed on the combined concentrates from the bench-scale continuous flotation circuit to attempt to recover a marketable Pb concentrate. A sample of the rougher concentrate (grading 6.8 pct Pb) was cleaned three times to produce a final Pb concentrate with a grade of 72 pct Pb. The cleaned Pb concentrate also contained, in percent, 0.95 Cu, 2.2 Fe, and 2.7 Zn. The overall cleaner recovery for Pb was 79 pct. The reagents in the cleaner test were sodium carbonate for pH adjustment (4.2 lb/st), sodium cyanide as a pyrite depressant (0.58 lb/st), a mixture of sodium diisobutyldithiophosphinate and triisobutylphosphine sulfide as the collector (0.02 lb/st), and a mixture of higher alcohols and hydrocarbon oil as the frother (0.1 lb/st). The collector was only added to the first cleaning stage and carried over in the subsequent cleaning stages. The concentrate grade of 72 pct Pb may be suitable for traditional smelter feed or as feed for a nontraditional hydrometallurgical treatment of Pb concentrates. Therefore, it may be possible to reprocess the National tailings pile and have a relatively clean final tailings containing, in parts per million, 400 Pb, 40 Cu, and 300 Zn and produce a somewhat marketable final Pb concentrate containing 72 pct Pb.

NEW LEAD BELT TAILINGS

Cominco's Magmont Tailings

Cominco American, Inc., Spokane, WA, operates the Magmont concentrator at Bixby, MO. The operation is a joint venture of Dresser Minerals Division of Dresser Industries, Houston, TX, and Cominco American (*6*). The Magmont operation has previously been detailed (*7*). In the past, the Bureau has characterized and investigated concentrates and tailings from a resource recovery standpoint (*8-9*). In this investigation the Bureau studied the tailings from the environmental standpoint of producing a relatively clean tailings product for a secondary use. This study may prove helpful if any of the tailings currently being generated come under tighter environmental regulations in the future.

The Magmont concentrator produces chalcopyrite, galena, and sphalerite concentrates by froth flotation. The tailings contain minor amounts of these minerals as well as minor amounts of siegenite $(Ni,Co)_3S_4$ and bravoite $(Ni,Co,Fe)S_2$ along with pyrite, marcasite, and the host rock dolomite (*8*). Mineralogical examination revealed only slight surface alterations of the galena particles. From samples taken by the Bureau, the tailings contain an average of, in percent, 0.18 Pb, 0.07 Cu, 2.47 Fe, 0.19 Zn, 0.011 Co, and 0.018 Ni. Tonnage estimates of the tailings at the Magmont operation are in the range of 20 million st. The tailings were sampled by the same method as the National tailings using the auger device. A typical screen analysis is shown in Table 2.

TEST PROCEDURES

Feed Preparation

The feed preparation was not as extensive as with the National tailings. Since the Magmont tailings were from a flotation concentrator, they had previously

TABLE 2. TYPICAL SCREEN ANALYSIS OF MAGMONT TAILINGS

Mesh	Size, µm	Wt Pct	Pct Pass	Chemical Analyses, Pct					
				Co	Cu	Fe	Ni	Pb	Zn
Plus 100............	+150	26.17	73.83	0.01	0.08	1.50	0.02	0.10	0.12
Minus 100 plus 150..	+106	16.71	57.12	.02	.16	2.10	.02	.09	.21
Minus 150 plus 200..	+74	11.85	45.27	.02	.22	3.20	.02	.10	.22
Minus 200 plus 270..	+54	19.21	26.06	.02	.10	3.50	.02	.12	.14
Minus 270 plus 400..	+37	.47	25.59	.01	.04	3.00	.02	.12	.12
Minus 400...........	-37	25.59	0	.02	.04	3.10	.03	.30	.23
Totals............	NAp	100	NAp	.01	.10	2.60	.02	.15	.18
				Distribution, Pct					
Plus 100............	+150	NAp	NAp	17.51	20.25	15.09	18.48	17.06	17.56
Minus 100 plus 150..	+106	NAp	NAp	16.76	25.85	13.48	15.72	9.80	19.61
Minus 150 plus 200..	+74	NAp	NAp	11.90	25.23	14.58	11.16	7.73	14.58
Minus 200 plus 270..	+54	NAp	NAp	19.28	18.58	25.83	18.08	15.02	15.03
Minus 270 plus 400..	+37	NAp	NAp	.31	.18	.54	.44	.36	.31
Minus 400...........	-37	NAp	NAp	34.24	9.90	30.49	36.13	50.03	32.90
Totals............	NAp	NAp	NAp	100	100	100	100	100	100

NAp Not applicable.

TABLE 3. CONDITIONS FOR THE REMOVAL OF LEAD FROM THE NEW LEAD BELT TAILINGS WITH FIVE FLOTATION STAGES

Reagent	Stage No.	Total lb/st	pH
Sulfide..................	1-4	0.60	9.5
Copper sulfate...........	1-4	.35	9.5
Potassium amyl xanthate..	1-4	.35	9.5
Tallow amine acetate.....	5	.87	10
MIBC[1] (frother)..........	1,2	.17	9.5

[1] MIBC Methyl isobutyl carbinol.

NOTE.--Sodium hydroxide was used to maintain pH throughout the test.

been milled to the liberation point. The only preparation given was a scrubbing period in the flotation cell prior to testing.

Rougher Froth Flotation

The standard flotation procedure for the Magmont tailings was the same as outlined for the National tailings and consisted of addition of additives, pH adjustment, addition of collector, addition of frother, and introduction of air. The normal conditioning time was 3 min per addition.

Initial flotation testing was performed with a tallow amine acetate collector at pH 10, but Pb recoveries were in the range of 40 to 50 pct and excessive weight was removed in the concentrates. The Cu and Zn recoveries were in the range of 50 to 60 pct; other amine collectors gave similar results. The addition of sodium sulfide to the amine system lowered the weight percent removed in the concentrate but did not dramatically increase Pb recovery. The addition of copper sulfate with the amines increased Zn recoveries, as expected, and boosted Cu recoveries to over 90 pct, but did little to improve Pb recoveries. It was clear from these tests that a multistage reagent scheme would need to be employed. A xanthate collector was added with sodium sulfide, in multiple flotation stages, ahead of the amine collector. This system improved Pb recoveries to >74 pct and lowered the Pb remaining in the reprocessed tailings to 600 ppm. The process only removed 9 pct of the original weight but recovered 95 pct of the Cu and 80 pct of the Zn values. Attempts were made to increase the recovery of the Pb values and thereby lower the Pb remaining in the final reprocessed tailings even further. Using this reagent scheme with copper sulfate added ahead of the xanthate and with the minus 400-mesh (-37 μm) fraction removed from the reprocessed tailings gave a final tailings containing, in parts per million, 400 Pb, 200 Cu, and 500 Zn. The final reprocessed tailings contained >75 pct of the original weight. This system recovered 85 pct of the Pb, 84 pct of the Cu, and 80 pct of the Zn. The conditions are given in Table 3.

While the Cu recovery dropped by 11 pct from the previously described test, the Pb recovery was increased by 10 pct using this reagent scheme. Keeping with the study's objectives of removing the Pb to below the target level of 500 ppm, these were acceptable results. The grade, combining all concentrates and the minus 400 mesh (-37 μm) fraction, was 0.72 pct Pb, 0.33 pct Cu, and 0.63 pct Zn. This combined concentrate was extremely low in grade (compared to other tests), but the reprocessed tailings containing 400 ppm Pb were actually the final product. Due to the low grades involved with the New Lead Belt tailings, no attempts were made to recover a marketable product from these concentrates.

It should be noted that the reprocessing of the southeast Missouri Pb tailings appears to be a site-specific problem where each tailings pile, like the original ores, will require a different reagent scheme. One possible reason is the difference in the degree of alteration of the sulfide minerals and their alteration products, especially galena, in the older piles as compared to the tailings of the New Lead Belt. Another possibility would be that the original ores, although containing much the same mineralization, had different mineral (or metal value) ratios. For instance, in the National pile, the Pb-Zn ratio was approximately 8:1, while in the Magmont tailings, the Pb-Zn ratio was approximately 1:1.

CONCLUSIONS

Froth flotation proved to be a viable method to reprocess both the Old and New Lead Belt tailings. The amount of lead remaining in the Old Lead Belt reprocessed tailings can be lowered to 400 ppm, with 94 pct of the original material remaining. The concentrates from the Old Lead Belt can be further upgraded to a marketable form, containing 72 pct Pb at a cleaner recovery of 79 pct. The sale of the Pb concentrates may help to offset a portion of the potential remediation costs, if necessary. The amount of lead remaining in the New Lead Belt reprocessed tailings can be lowered to 400 ppm, with >75 pct of the original material remaining. Much like the original lead ores, the reprocessing of the tailings appears to be site specific.

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