RI 9277

REPORT OF INVESTIGATIONS/1989

National Mine Health & Safety Academy
Informational Services Branch
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Recovery of Lead and Silver From Plumbojarosite by Hydrothermal Sulfidation and Chloride Leaching

By N. P. Gallagher and K. P. V. Lei

BUREAU OF MINES

UNITED STATES DEPARTMENT OF THE INTERIOR

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Library of Congress Cataloging in Publication Data:

Gallagher, N. P.
Recovery of lead and silver from plumbojarosite by hydrothermal sulfidation and chloride leaching / by N. P. Gallagher and K. P. V. Lei.

p. cm. – (Report of investigations / United States Department of the Interior, Bureau of Mines; 9277)

Bibliography: p. 9

Supt. of Docs. no.: I 2823:9277.

1. Lead–Metallurgy. 2. Silver–Metallurgy. 3. Plumbojarosite. 4. Sulfidation. 5. Leaching. I. Lei, K. P. V. (Kenneth P. V.). II. Title. III. Series: Report of investigations (United States. Bureau of Mines); 9277.

TN23.U43 [TN770] 622 s–dc20 [669'.4] 89-600179

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	min	minute
cm	centimeter	mL	milliliter
g	gram	mol/L	mole per liter
g/L	gram per liter	pct	percent
h	hour	ppm	part per million
L	liter	psig	pound (force) per square inch, gauge
lb/st	pound per short ton	rpm	revolution per minute
μm	micrometer	tr oz/st	troy ounce per short ton
mg/L	milligram per liter		

RECOVERY OF LEAD AND SILVER FROM PLUMBOJAROSITE BY HYDROTHERMAL SULFIDATION AND CHLORIDE LEACHING

By N. P. Gallagher[1] and K. P. V. Lei[2]

ABSTRACT

The U.S. Bureau of Mines investigated a hydrothermal sulfidation (HTS) and chloride leaching procedure to recover lead and silver from plumbojarosite. The plumbojarosite, generated during pressure-oxidative leaching of zinc concentrate in a commercial operation, contained, in percent, 25.4 Pb, 18.8 Fe, 1.4 Zn, 29.5 sulfate (SO_4^{2-}), 3.2 elemental sulfur (S°), 13.4 total S, and 7.4 tr oz/st Ag. The HTS was conducted in an autoclave and converted the plumbojarosite into amorphous iron oxide and liberated the Pb and Ag as sulfides (S^{2-}). The best operating conditions were 50 g of plumbojarosite and 7 pct S° (2.04 g S° added) in 500 mL of 2.0-mol/L NH_3 at 150° C for 1 h. The sulfidation product contained, in percent, 37 Pb, 28 Fe, 2 SO_4^{2-}, 7 total S, and 11.2 tr oz/st Ag. Flotation of the sulfidation product was conducted at pH 3 with 15-pct pulp density and 2.4 lb/st of isopropyl xanthate as collector. The flotation concentrate contained 43 pct Pb, 25 pct Fe, and 13 tr oz/st Ag. $FeCl_3$ leaching of the sulfidation product in a resin kettle extracted 98 pct Pb and 97 pct Ag from the sulfidation product, and HCl-O_2 leaching in a shaker glass bottle extracted 99 pct Pb and 98 pct Ag. Both leach residues met the Environmental Protection Agency's Toxicity Characteristic Leaching Procedure (EPA-TCLP) standards for nonhazardous wastes. The HTS and chloride leaching procedure is a potentially effective method for detoxification of plumbojarosite-bearing residue from the zinc pressure-leaching circuit.

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INTRODUCTION

A primary method for iron control in hydrometallurgy is the precipitation of iron in the form of alkali jarosite or ammonium jarosite or a mixture of jarosite members (*1-3*).[3] The general formula of jarosite is $[MFe_3(SO_4)_2(OH)_6]$ in which M represents H_3O^+, Na^+, K^+, Rb^+, Ag^+, NH_4^+, $Pb_{0.5}^{2+}$, and $Hg_{0.5}^{2+}$. Plumbojarosite and hydronium jarosite are formed in the commercial oxidative acid pressure-leaching process for zinc sulfide concentrates (*2, 4-6*). The precipitates contain environmentally objectionable metals, such as Pb, Hg, or Ag, which must be treated to effect a safe disposal. Because of substitution by these metals into the jarosite lattice, recovery of metals requires the destruction of the jarosite mineral to release the metals (*7-8*). Silver cannot be leached from argentojarosite by direct cyanidation (*9*), and lead cannot be extracted from plumbojarosite by solvents, such as sodium chloride (NaCl) or ammonium acetate ($NH_4C_2H_3O_2$) (*10*). The destruction of jarosite has been accomplished by concentrated acid leaching or converting the jarosite to hematite or goethite with bases (*11*). The convertion in basic solutions is often referred to as hydrothermal conversion (HTC). The released metals may be recovered by conventional techniques, such as solvent extraction, precipitation, ion exchange, or electrolysis.

Jarosite residues formed in the zinc pressure-leaching circuit are being dealt with in a number of ways (*3*): (1) redissolve the residue in the calcine-leaching circuit, (2) impound the residue, and (3) upgrade the residue separate from the calcine leach circuit, and process through smelting or hydrometallurgical processes. The hydrothermal sulfidation procedure investigated in this U.S. Bureau of Mines report belongs to the third category. The procedure assumes that the lead and silver in the jarosite can be converted to metal sulfides during the HTC of the jarosite phase in the presence of elemental sulfur. The metals are recovered by sulfide flotation or by chemical leaching.

The HTC of jarosite consists of decomposing the jarosite into iron oxide, metal sulfates, and sulfuric acid (H_2SO_4). The H_2SO_4 is neutralized by a base, e.g.,

$$2MFe_3(SO_4)_2(OH)_6 \rightarrow 3Fe_2O_3 + 3H_2O + M_2SO_4 + 3H_2SO_4, \quad \text{(A)}$$

$$3H_2SO_4 + 6OH^- \rightarrow 3SO_4^{2-} + 6H_2O, \quad \text{(B)}$$

where M = Na, K, NH_4, $Pb_{0.5}$, Ag, etc.

The elemental sulfur added with the jarosite disproportionates under the basic conditions to form sulfidizing species, such as S^{2-}, HS^-, and $S_2O_3^{2-}$:

$$4S^\circ + 8OH^- \rightarrow SO_4^{2-} + 3S^{2-} + 4H_2O, \quad \text{(C)}$$

$$3S^\circ + 6OH^- \rightarrow SO_3^{2-} + 2S^{2-} + 3H_2O \quad \text{(D)}$$

$$4S^\circ + 4OH^- \rightarrow S_2O_3^{2-} + 2HS^- + H_2O \quad \text{(E)}$$

Lead and silver sulfates sulfidize by S^{2-}, HS^-, and $S_2O_3^{2-}$ are as follows:

$$PbSO_4 + S^{2-} \rightleftharpoons PbS + SO_4^{2-}, \quad \text{(F)}$$

$$Ag_2SO_4 + S^{2-} \rightleftharpoons Ag_2S + SO_4^{2-} \quad \text{(G)}$$

$$PbSO_4 + OH^- + HS^- \rightarrow PbS + SO_4^{2-} + H_2O \quad \text{(H)}$$

$$Ag_2SO_4 + OH^- + HS^- \rightarrow Ag_2S + SO_4^{2-} + H_2O \quad \text{(I)}$$

$$PbSO_4 + 2OH^- + S_2O_3^{2-} \rightarrow PbS + 2SO_4^{2-} + H_2O \quad \text{(J)}$$

$$Ag_2SO_4 + 2OH^- + S_2O_3^{2-} \rightarrow Ag_2S + 2SO_4^{2-} + H_2O \quad \text{(K)}$$

This Bureau report describes the results of a bench-scale investigation of an HTS procedure for converting plumbojarosite into iron oxide and lead and silver sulfides with elemental sulfur in an ammonia solution. The results for the recovery of Pb and Ag by preparing a Pb-Ag flotation concentrate from the sulfidation product and from chloride leaching of the sulfidation product are reported.

MATERIALS, EQUIPMENT, AND PROCEDURES

The plumbojarosite sample was obtained from the zinc concentrate pressure-leaching circuit of a commercial zinc producer. The sample had been subjected to flotation to remove elemental sulfur before it was shipped, and it contained approximately 24 pct moisture. The jarosite was stored, as received, until needed, at which time the sample was slurried in deionized water, filtered, and dried at 100° C for 24 h. The composition of the sample was, in percent, 18.8 Fe, 25.4 Pb, 1.4 Zn, 29.5 SO_4^{2-}, 3.2 S°, 13.4 total S, and 7.4 tr oz/st Ag. It also contained 0.02 pct Cu. X-ray diffraction analysis showed that the major component of the jarosite was plumbojarosite, the minor component was anglesite ($PbSO_4$), and the trace component was silicon dioxide (SiO_2). From lead, iron, and sulfate analyses, and assuming that the lead compounds were plumbojarosite $[PbFe_6(SO_4)_4(OH)_{12}]$ and $PbSO_4$, the composition was approximately 63 pct plumbojarosite, 20 pct $PbSO_4$, and 17 pct other materials, such as zinc sulfide ZnS, SiO_2, etc.

The elemental sulfur was reagent grade. The aqueous NH_3 was made by diluting reagent-grade concentrated ammonium hydroxide (NH_4OH) with deionized water. In this U.S. Bureau of Mines report, NH_3 concentrations refer to the combined concentrations of $NH_{3(aq)}$, $NH_4OH_{(aq)}$, and $NH_4^+{}_{(aq)}$. The collector used in the flotation was Aero[4] 343 (isopropyl xanthate), while frothing action was provided by Dowfroth 250. The collector and frother were prepared with distilled water (H_2O).

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[3] Italic number in parentheses refer to items in the list of references at the end of this report.

All HTS experiments were conducted in a Parr 2-L titanium autoclave equipped with a six-bladed turbine impeller operated at a constant stirring rate of 750 rpm. Heating was supplied by a wraparound heating mantle, while cooling for reaction quenching and temperature control was provided by internal water-cooled coils. A time proportioning temperature controller was used to control the autoclave temperature to ±2° C. The reactor slurry was filtered on a 12.5-cm Buchner vacuum filter using Whatman No. 3, No. 5, or No. 50 paper. Flotation experiments were conducted in a 50-g capacity flotation cell.

The plumbojarosite, elemental sulfur (if added), and basic solution were charged into the glass-lined autoclave, which was sealed. The total elemental sulfur content in the sulfidation feed, in weight percent, was determined by the following formula:

$$\frac{\text{S}^\circ \text{ in feed } + \text{ S}^\circ \text{ added}}{\text{Feed added } + \text{ S}^\circ \text{ added}} \times 100 = \begin{matrix}\text{percent S}^\circ \text{ content in} \\ \text{sulfidation feed.}\end{matrix}$$

The autoclave was purged twice with nitrogen to remove residual air. After purging, the reactor was pressurized to approximately 20 psig N and heated to the reaction temperature. In the range of 100° to 150° C, the difference in heat-up time was less than 6 min. The final pressure of the reactor at the operating temperatures varied from 80 to 135 psig. The variation was due to the changes in vapor pressures of aqueous solutions of NH_3 used in the sulfidation. After reaching the reaction temperature, the reaction was allowed to proceed for the desired time, after which the reactor was quenched in an ambient temperature H_2O bath for approximately 5 min. The contents were filtered, and the solid residue dried at 100° C for at least 12 h.

Flotation of the HTS product was performed by adding the product and H_2O to the flotation cell. After conditioning for approximately 5 to 10 min, the collector was added after conditioning and the flotation was begun by sparging nitrogen into the bottom of the cell. Sufficient gas and frother were added to maintain bubble generation until little collection was observed visually (20 to 30 min). After flotation, both concentrate and tailing were filtered and dried in air at 100° C.

$FeCl_3$ leaching (*12-13*) of the sulfidation product was conducted with 50 g of sulfidation product in 500 mL of solution containing 110 g NaCl and 35 g of $FeCl_3$ at 93° C in a 1-L resin kettle equipped with a H_2O condenser. The heat and stirring (650 to 780 rpm) were provided by a hot plate-stirrer. After leaching for 1 h, the slurry was filtered at 75° C, and the filter cake was transferred to the kettle and releached with 500 mL of solution containing 50-g/L NaCl at 90° C for 2 h. The leached residue was separated by filtration at 75° C, washed with approximately 250 mL of solution containing 50-g/L NaCl at 80° C, and finally rinsed with approximately 300 mL of H_2O at 70° C. The final filter cake was dried at 100° C for at least 6 h.

HCl-O_2 leaching (*14*) was conducted with 30 g of the sulfidation product in 100 g H_2O, 50 g anhydrous $CaCl_2$, 9.2 g concentrated HCl (~37-pct HCl), 115° C, 40 psig O_2, and 24 h in a 0.5-L rubber-stoppered glass reaction bottle equipped with inlets for thermocouple and O_2. A heating mantle with a temperature controller was used to maintain the leaching temperature. Stirring of the slurry was provided by a Parr shaker-type hydrogenator equipped with a rocking mechanism operated at 225 cycles per minute. After leaching, the reactor was cooled to 22 ° C and the slurry was filtered. The filter cake was washed with approximately 1 L of solution containing 200-g/L NaCl and 1 L of H_2O before it was dried at 100° C for at least 6 h.

The EPA-TCLP (*15*) was used to determine the mobility of the heavy and toxic metals in the $FeCl_3$ and HCl-O_2 leach residue. Metals in both the solutions and solids were analyzed by inductively coupled plasma spectroscopy. Sulfate, S^{2-}, and S° were determined by wet chemical analysis, and precious metals by fire assay. X-ray fluorescence, x-ray diffraction, and scanning electron microscopy (SEM) analyses were used to examine selected solid samples.

RESULTS AND DISCUSSION

Data from the HTS experiments were analyzed in two ways to evaluate the process. The first, percent conversion, was defined as the difference between the initial SO_4^{2-} in the feed jarosite and that in the HTS product divided by the initial SO_4^{2-} in the feed jarosite. Thus, the percent conversion refers to the total SO_4^{2-} release from the feed material. The second consisted of evaluating the fate of elemental sulfur in the HTS. This evaluation determined the efficiency of using disproportionation of S° as a source of sulfide (S^{2-}). This involved calculating the percent of elemental sulfur reacting to form S^{2-}, SO_4^{2-}, and other aqueous S species "S" from SO_4^{2-}, S°, and total S analysis of both liquids and solids. In this report,"S" means aqueous sulfur species besides SO_4^{2-}, such as thiosulfate ($S_2O_3^{2-}$), sulfite (SO_3^{2-}), and dithioate ($S_2O_6^{2-}$).

Preliminary tests were conducted to determine approximate conditions under which plumbojarosite would transform to iron oxide and other products. The results indicated that temperatures above 110° C were necessary to obtain a satisfactory conversion rate, and that the neutralizing base had a profound effect on the conversion products involving lead and sulfur. Using NaOH, the predominant phases in the conversion product were amorphous oxides with very low concentrations (<1 pct) of SO_4^{2-} and S^{2-}. Using NH_3, however, the conversion products consisted of an amorphous iron oxide phase with higher SO_4^{2-} and S^{2-} compositions and galena. Very little elemental sulfur remained after the conversion. Although no silver sulfide (Ag_2S) was identified in the conversion product, silver was recovered as mixed metal sulfide concentrate by flotation.

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[4] Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

Additional tests were conducted to determine the effects of conversion time, temperature, and ammonia concentration on the hydrothermal sulfidation of the plumbojarosite.

EFFECT OF TIME ON SULFIDATION

A series of tests were conducted to determine the effect of time on the HTS and disproportionation of elemental sulfur in the jarosite. The results are displayed in table 1. The reaction conditions were 50 g jarosite, 500 mL of solution containing 1.0-mol/L NH_3, 150° C, and 1 h. At time zero, which is the time the reactor reached 150° C, the percent conversion (SO_4^{2-} release) was 43.8 pct. Thus, the jarosite was already partially decomposed and the elemental sulfur was already disproportionated to SO_4^{2}, S^{2-}, and "S". As the HTS process proceeded, the conversion quickly reached >90 pct while the "S" continued to react with the solid phase to form metal sulfides.

Table 1.–Effect of time on HTS conversion and disproportionation of S° in jarosite

(Reaction conditions: 50 g jarosite, 500 mL 1.0-mol/L NH_3, 150° C, and 1 h)

Time, h	Conversion, pct	Disproportionation of S°, pct		
		SO_4^{2-}	S^{2-} (s)	"S" (l)[1]
0	43.8	31.7	27.2	41.1
0.25	83.9	32.5	43.3	24.2
0.5	91.7	31.8	52.9	15.3
1	93.3	34.1	56.2	9.70
2	94.6	35.5	56.8	7.70

l Liquid.
s Solid.
[1] Aqueous sulfur species besides SO_4^{2-}.

The relative rates of metal S^{2-} formation and SO_4^{2-} release from the solid are indicated in figure 1 in which the concentrations of S^{2-} and SO_4^{2-} in the HTS product are plotted versus time. Examination of the slopes of the curves early in the HTS (within 30 min) shows that the SO_4^{2-} release from the jarosite was initially rapid, while the sulfidation of the metal sulfates occurred at a moderate rate. At times >1/2 h, both rates approached steady state, and the bulk of sulfidation and conversion were accomplished within 1 h at 150° C.

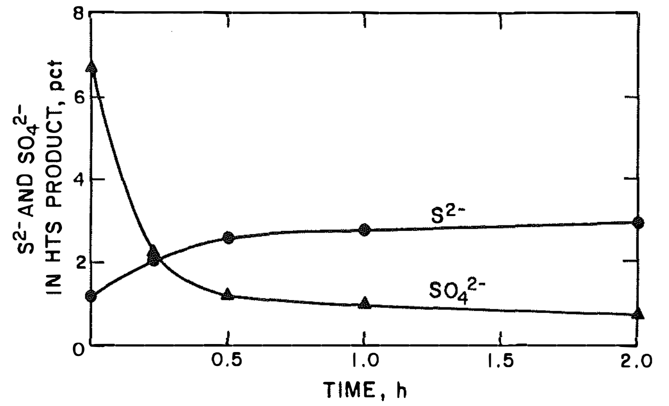

Figure 1.–Variation of sulfide and sulfate content of HTS product with time at 150° C.

EFFECT OF ELEMENTAL SULFUR CONTENT ON SULFIDATION

The effect of elemental sulfur content in the feed on sulfidation was investigated. The reaction conditions were 50 g jarosite, 500 mL of solution containing 2.0-mol/L NH_3, 150° C, and 1 h. Figure 2 shows the sulfur disproportionation stoichiometry by plotting the percentage of S° reacted to form S^{2-}, SO_4^{2-}, and "S" versus elemental sulfur content in the feed. Sulfide formation was constant and was not influenced by the elemental sulfur content. At low elemental S concentrations, S disproportioned largely to SO_4^{2-}, whereas at higher elemental S concentrations, the reaction favored the formation of "S".

Figure 3 shows the S^{2-} and SO_4^{2-} concentrations of the HTS product as a function of elemental sulfur content of the jarosite mixture. As the elemental sulfur concentration increases, the sulfide concentration increases substantially while the sulfate concentration decreases slowly. Stoichiometric PbS formation would result in a S^{2-} concentration of 5.7 pct in a product containing approximately 37 pct Pb. Although higher S^{2-} contents are possible at high elemental sulfur concentrations, SEM examination showed that galena particles in the HTS products prepared with 15 pct S° intergrew with the amorphous iron oxide phase much more than those prepared with 7 pct S°. As evidenced in figure 3, an elemental sulfur concentration of 7 pct results in a sulfide concentration in stoichiometric proportion to the lead content. Further testing was conducted with elemental sulfur concentrations fixed at 7 pct.

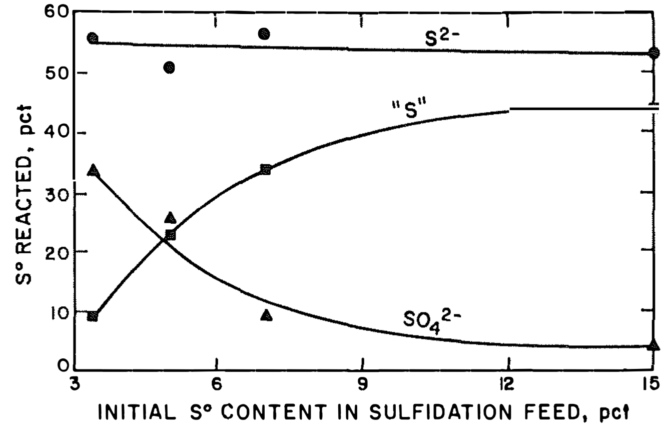

Figure 2.–Disproportioning stoichiometry of S° as function of initial S° content in sulfidation feed.

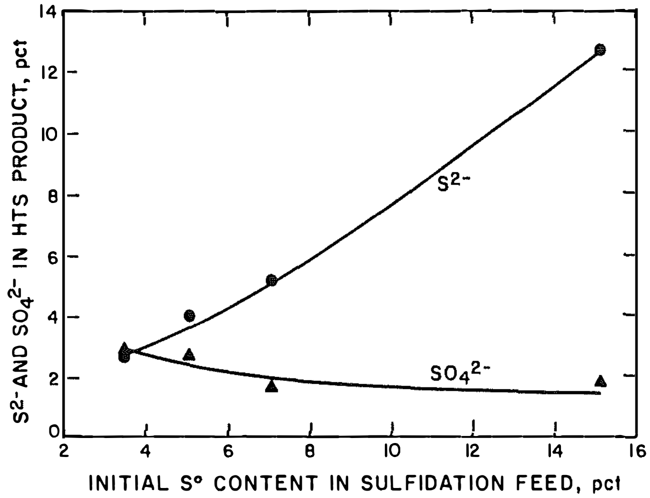

Figure 3.–Variation of sulfide and sulfate content of HTS product with initial S° content in sulfidation feed.

EFFECT OF TEMPERATURE AND NH_3 CONCENTRATION ON SULFIDATION

The extent of the elemental sulfur reaction and the disproportionation stoichiometry at NH_3 concentrations from 1.0 to 2.0 mol/L with respect to temperature is shown in figure 4. The HTS conditions were 50 g jarosite, 500 mL NH_3 solution, 7 pct total elemental sulfur in the jarosite, and a reaction time of 1 h. As the temperature increased, the extent of sulfur reaction increased at all three NH_3 concentrations. Figure 4 shows the reaction was completed at 150° C and independent of the NH_3 concentrations. However, the amount of reacted S° disproportionated to S^{2-} was the highest and was 44 pct with 2.0-mol/L NH_3 at 150° C, compared with 35 and 40 pct with 1.0- and 1.5-mol/L NH_3, respectively.

Selected samples of the HTS products from 2.0-mol/L NH_3 and 110° to 150° C were examined by x-ray diffraction and SEM. The constituents of the HTS products were identified as galena, amorphous oxides, and hematite. Figure 5*A* shows a X 2,000 SEM photomicrograph of the jarosite sample loosely mounted showing the relationship of (1) flakelike plumbojarosite particles with respect to (2) the $PbSO_4$ particles and (3) SiO_2 particles. The plumbojarosite particles are of a fairly uniform size (less than 10 μm), while the $PbSO_4$ particles are less uniform in size and generally less than 5 μm in size. Figures 5*B*, 5*C*, and 5*D* show X 2,000 SEM photomicrographs of HTS products obtained at 110° C, 130° C, and 150° C, respectively. The 110° C conversion product was only partially converted with distinct plumbojarosite platelets still observable. At 130° C, the grain structure has deteriorated to the amorphous iron oxide phase with intermixing of small galena particles. Product converted at 150° C shows further deterioration in grain structure. This deterioration of jarosite phase from larger grains to an aggregate of much smaller grains was also observed by Kunda and Veltman (*11*) during the hydrothermal conversion of sodium and ammonium jarosite. The galena particles in 5*D* are larger in size and indicate that grain growth of galena via a dissolution and precipitation mechanism might be occurring at the higher temperatures.

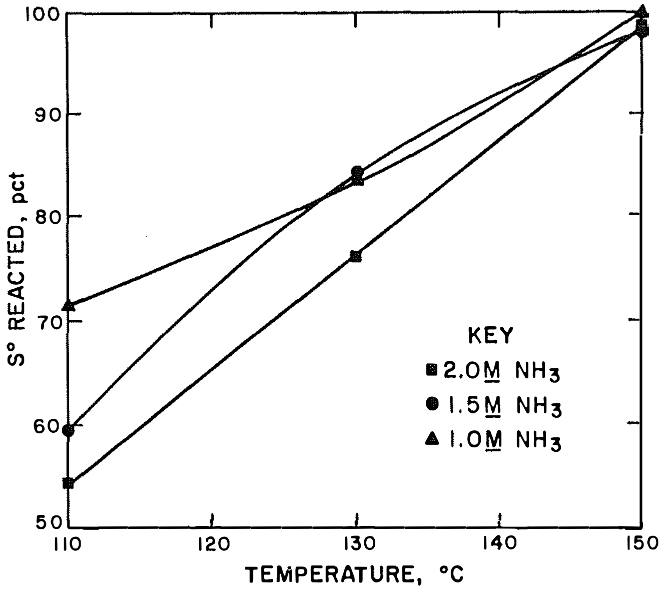

Figure 4.–Reaction of S° as function of temperature at different NH_3 concentrations.

Based on the results, the best conditions for HTS are 50 g jarosite containing 7 pct S°, 500 mL of 2.0-mol/L NH_3, 150° C, and a reaction time of 1 h. This results in a product with a mole ratio of lead to sulfide of approximately 1.0.

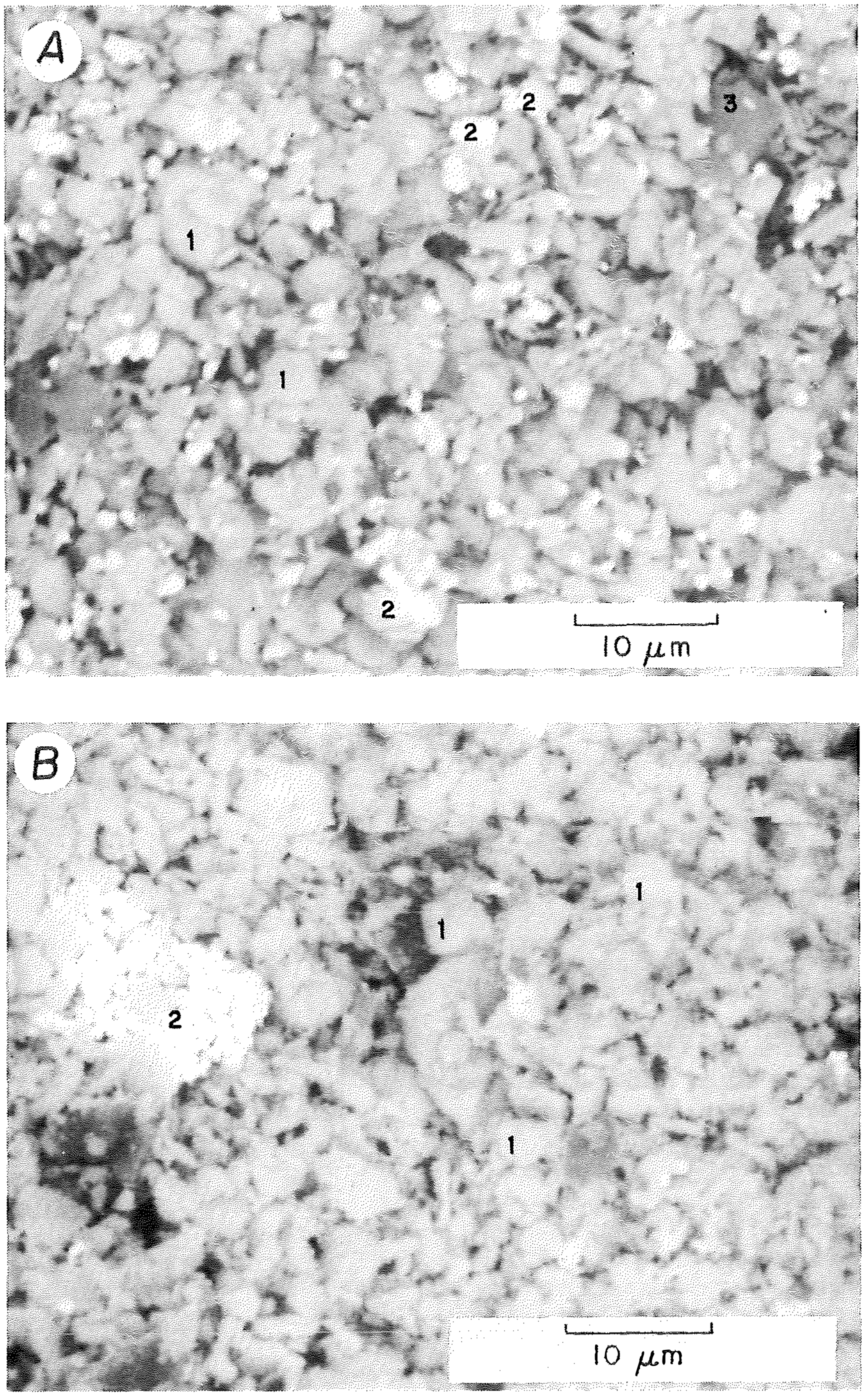
A
2
2
3
1
1
2
10 μm
B
1
1
2
1
10 μm

Figure 5.–SEM photomicrographs of plumbojarosite and HTS products (X 2,000). *A*, Plumbojarosite: 1–distinct plumbojarosite particles (<10 m), 2–$PbSO_4$ (<5 μm), 3–SiO_2; *B*, HTS product at 110° C: 1–plumbojarosite particle still discrete, 2–$PbSO_4$.

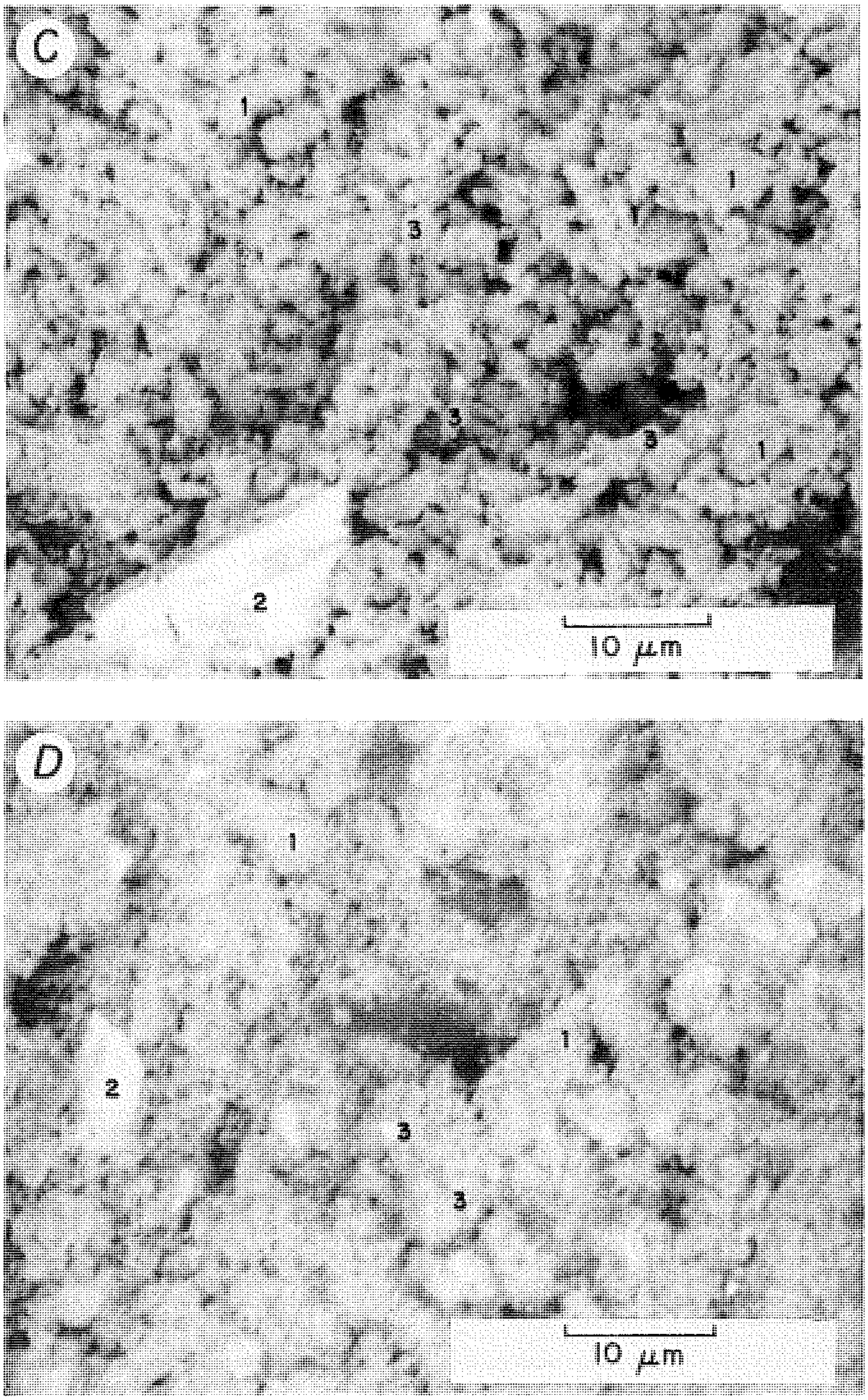

Figure 5.–SEM photomicrographs of plumbojarosite and HTS products (X 2,000) –Continued. *C*, HTS product at 130° C showing deterioration: 1–plumbojarosite phase, 2–$PbSO_4$, 3–galena particles; *D*, HTS product at 150° C showing further deterioration: 1–plumbojarosite phase, 2–$PbSO_4$, 3–galena particle.

FLOTATION OF SULFIDATION PRODUCT

The small particle size of the conversion product, in conjunction with the surface charge difference between the iron oxide phase and lead sulfide, would present sliming problems. However, preliminary experiments showed that acidic pH conditions inhibited the sliming due to a dispersive effect between galena, point of zero charge (pzc) = 3, and iron oxide, pzc = 6.5. A collector addition of 200 mg/L Aero 343 and a pulp at 15 pct solid were needed for effective concentration of sulfides.

A series of flotation tests were performed on the HTS products obtained under the following conditions: 100 g of jarosite, 1 L 2.0-mol/L NH_3, 150° C, 7 pct S°, and 1 h. Flotation conditions were 200 mg/L Aero 343, 170 g/L of sulfidation feed, and pH 3. A mass ratio of concentrate to tailing of approximately 2.4:1 resulted from the flotation. The recovery for lead ranged from 73 to 93 pct, and 80 to 94 pct for silver. The average grade of concentrate produced was 43 pct Pb, 13 tr oz/st Ag, and 25 pct Fe.

Because the tailing contained approximately 22 pct Pb, 35 pct Fe, and 5 tr oz/st Ag, recycle of the tailing to the HTS step was investigated as a means of decreasing the lead and silver losses to the tailing. The conversion conditions were the same as above, but included the tailing from the flotation step. The results for two cycles of sulfidation-flotation of the tailing leading to three concentrates and one final tailing showed that the grade of concentrate did not significantly change compared with that produced in the single cycle HTS-flotation. The final tailing contained 16 pct Pb, 35 pct Fe, and 4 tr oz/st Ag. The overall results indicate that the flotation technique was unable to achieve high recovery of lead and silver and produce a high-grade Pb-Ag concentrate from the HTS product.

CHLORIDE LEACHING OF SULFIDATION PRODUCT

Because flotation was not successful in preparing a high-grade Pb-Ag concentrate from the sulfidation product, $FeCl_3$ (*12-13*) and $HCl\text{-}O_2$ (*14*) leaching were investigated for extracting the silver and lead from the sulfidation product. The $HCl\text{-}O_2$ leaching procedure is capable of producing a higher redox potential than the $FeCl_3$ leaching procedure, which could facilitate the extraction of silver. Table 2 compares the analysis of the sulfidation product and the leach residue and indicates that both chloride procedures were suitable for recovering high amounts of lead and silver from the sulfidation product. The extractions of lead and silver were 98 and 97 pct, respectively, by the $FeCl_3$ procedure, and 99 and 98 pct, respectively, by the $HCl\text{-}O_2$ procedure.

Table 2.–Analysis of sulfidation product and residue from chloride leaching, percent

Element	Sulfidation product	$FeCl_3$ leach residue[1]	$HCl\text{-}O_2$ leach residue[2]
Pb	37.6	1.6	0.9
Fe	29.5	50.0	52.1
Zn	.80	.80	.70
Sulfate (SO_4^{2-})	2.4	1.6	1.4
Sulfide (S^{2-})	6.1	1.7	1.4
Sulfur (S°)	ND	7.6	7.3
Ag . . Tr oz/st	11.2	0.8	0.5

ND Not determined.

[1] 18.8 g residue obtained by leaching 50 g sulfidation product in 500 mL solution containing 110 g NaCl and 35 g $FeCl_3$ at 93° C for 1 h.

[2] 21.8 g residue obtained by leaching 50 g sulfidation product in 100 g water containing 5 g $CaCl_2$, 9.2 g 37-pct HCl, 115° C, 40 psig for 24 h.

Both residues contained elemental sulfur and hematite as major constituents and silica as the minor constituent. The residues from both chloride leachings were subject to the EPA-TCLP. The extracts contained less than 1.0 ppm each for As, Cd, Pb, and Ag, and satisfied the EPA regulatory limit for the inorganic contaminants (*15*), and the residue can be classified as a nonhazardous residue for disposal. EPA-TCLP tests conducted on the plumbojarosite showed that the extracts contained 180 to 190 ppm Pb.

SUMMARY AND CONCLUSIONS

It is possible to decompose and stabilize the iron found in plumbojarosites as amorphous iron oxide, while concomitantly recovering the lead and silver present. The HTS procedure took advantage of the fact that elemental sulfur present in the plumbojarosite disproportionates to form lead and silver sulfides in ammonia solutions at elevated temperatures. Flotation was not effective for producing a high-grade concentrate from the HTS products. At least 98 pct of the lead and 97 pct of the silver were extracted from the sulfidation product by oxidative chloride leaching. The results demonstrated that the hydrothermal sulfidation combined with leaching is a potentially effective method for detoxification of plumbojarosite-bearing residue from the zinc pressure-leaching circuit.

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