GOLD AND SILVER EXTRACTION FROM COMPLEX SULFIDE WASTES

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Summary

The present study has demonstrated the application of different leaching techniques in the extraction of precious metals from extreme low grade materials such as zinc and lead flotation tailings. The bacterial leaching preceeding thiourea treatment seem to expose precious metal inclusions from the sulfide matrix. Preliminary results indicate gold extractions by thiourea near 60% for samples pretreated with microorganisms and about 40% for samples not treated by bacteria.

In another series of experiments, the received tailings were prewashed with HCl and subsequently leached with thiourea. Extractions of gold as high as 81% have been obtained. Thiosulfate leaching at 50°C resulted in highest extraction which was approximately 95%. Two step continuous countercurrent process has been proposed for the premined and preground flotation tailings. The efficiency of this approach has been demonstrated. The relative economic viability of three methods: cyanidation, thiourea leaching, and thiosulfate leaching has been discussed on basis of preliminary data obtained in this investigation and in others. Those preliminary data are encouraging but much work remains to be done in order to optimize and scale up the processes to pilot plant level.

Introduction

The involvement of the bacterial leaching in the precious metal extraction is a new development (1-10); the same can be said about the utilization of thiourea (11-22) and thiosulfates (23-26). The role of microorganisms in these processes is to liberate the finely disseminated gold and silver inclusions from the sulfide mineral matrix (10, 27, 28). Pyrite is always associated with flotation residues of metal sulfides. Consequently the microbes oxidize metal sulfides to their respective sulfates (29, 30):

$$MS + 2O_2 \xrightarrow{bacteria} MSO_4 \qquad [1]$$

This can be considered as the direct way of bacterial action. The indirect way of bacterial activity (29, 31) can be expressed by the following reaction:

$$MS + Fe_2(SO_4)_3 \longrightarrow MSO_4 + 2\,FeSO_4 + S^o \qquad [2]$$

The ferrous sulfate and elemental sulfur produced in this process will be oxidized by the bacteria to yield ferric sulfate:

$$2\,FeSO_4 + 1/2O_2 + H_2SO_4 \xrightarrow{bacteria} Fe_2(SO_4)_3 + H_2O \qquad [3]$$

and

$$S^o + 1.5O_2 + H_2O \xrightarrow{bacteria} H_2SO_4 \qquad [4]$$

Ferric sulfate is known to be a powerful oxidation agent (15, 31, 32). In the bacterial leaching process probably both mechanisms: direct and indirect, are present.

As the bacterial leaching is completed and precious metals are liberated, they can be reached easily by the thiourea lixiviant, the reaction with which (12, 14) can be expressed by

$$2\,Au + 2CS(NH_2)_2 + (CS)_2(NH_2)_2(NH)_2 + 2H^+ = 2Au[CS(NH_2)_2]_2^+ \qquad [5]$$

One of the reactants in the above reaction is formamidine disulfide which itself is a product of the partial oxidation of thiourea, (simultaneously occuring).

If ferric ion is present (12), then

$$Au + Fe^{3+} + 2\,CS(NH_2)_2 = Au[CS(NH_2)_2]_2^+ + Fe^{2+} \qquad [6]$$

In reactions [5] and [6] the thiourea - gold complex is a cationic species which forms according to reaction

$$Au + 2CS(NH_2)_2 = Au[CS(NH_2)_2]_2^+ + e^- \qquad [7]$$

Thiourea losses occur because of parasitic side-reactions, such as successive oxidation, the first step of which (12, 14) is

$$2\,CS(NH_2)_2 = (CS)_2(NH_2)_2(NH)_2 + 2H^+ + 2e^- \qquad [8]$$

Silver reacts with thiourea as follows:

$$2\,Ag + 2H^+ + 1/2O_2 + 6CS(NH_2)_2 = 2\,Ag[CS(NH_2)_2]_3^+ + H_2O \qquad [9]$$

and silver sulfide (14) reacts in the presence of ferric ion:

$$Ag_2S + 2\ Fe^{3+} + 6\ CS(NH_2)_2 = 2\ Ag[CS(NH_2)_2]_3^+ + Fe^{2+} \qquad [10]$$

Ferric ion (15) apparently helps improving the kinetics of silver dissolution in thiourea.

As far as thiosulfate dissolution is concerned, Lenher (36, 37) postulates the formation of a double sulfate of gold

$$X_2S_2O_3 + Au + 1/2\ H_2O + 1/4\ O_2 = XAuS_2O_3 + XOH \qquad [11]$$

where X either NH_4^+ or Na^+.

One should note that reaction [11] results in creating more and more basic environment. Simple thiosulfates plus basic hydroxides form, due to the reaction of a aqueous solution of thiosulfate with silver. Other areas of a similar problem were investigated by (1, 23-26).

The purpose of this study was to explore new possibilities in exploiting premined waste materials.

The results are preliminary, but if reconfirmed, could have a considerable economic significance.

Experimental

The detailed description of experimental conditions and materials has been given elsewhere (33), however for a better understanding and follow up the main points can be reiterated.

The mineral studied was Pecos Mine flotation tailings (34) containing, according to authors' analysis around 1.75 g per metric ton gold and 22.5 g per metric ton silver. The bacteria used was a pure strain of Thiobacillus ferrooxidans isolated from acid mine drainage (8), maintained on a 9K nutrient solution. All reagents were described in (33). Gold was analysed via the MIBK method on a Perkin Elmer AA Spectrometer, as outlined by (35). Silver was analysed on the same apparatus, directly from the aqueous solutions.

The reactors were standard glass reactors supplied with temperature, pH and rpm controls and with a condenser.

Bioleaching experiments followed by thiourea leaching, hydrochloric acid preleachings followed by thiourea lixiviation, and thiosulfate leachings (both in a single step batch reactor, and in a double step continous countercurrent system) were investigated. Some carbon (Calgon GRC-11) adsorption experiments were also included in the study.

Results

The gold recovery by thiourea leaching amounted to 16.5% initially. When bacterial preleach was applied and DuPont Oxone added to the thiourea leachant, gold extractions reached 56% and silver extraction rose from 42% to 67%. The optimum Oxone concentration was 3.3 gdm^{-3}.

Table 1 presents the results of this part of the investigation.

Table 2, 3 and 4 illustrate the second part of the study. Gold extraction is plotted versus time at different thiourea concentrations in Table 3. Table 2 gives the effect of acid concentration in the thiourea leachant, and

Table 1.

Extraction of Gold and Silver by Thiourea with Bacterial Preleach

Time (min)	Extraction (%)							
	TU only		TU + OX		BLP + TU		BPL + TU + OX	
	Au	Ag	Au	Ag	Au	Ag	Au	Ag
30	5.6	26.4	8.8	35.6	21.2	35.6	28.0	38.4
60	6.6	36.6	14.3	42.6	31.2	45.6	43.2	49.6
120	9.9	38.4	19.2	46.8	37.6	53.6	50.4	61.6
180	13.2	41.4	22.4	47.2	39.2	54.4	53.6	65.6
240	16.5	42.1	23.0	47.7	39.5	55.2	56.0	67.2

BPL= Bacterial preleach; TU=thiourea 0.5 M; OX= Oxone 3.3 gdm^{-3};Temp.=25^{o}C;

Table 2.

Effect of HCl Concentration on Gold Extraction by Thiourea

HCl moldm^{-3}	Extraction (%)	HCl moldm^{-3}	Extraction (%)
0.0	0	0.225	60
0.05	0	0.25	60
0.10	1	0.30	55
0.15	13	0.35	49
0.20	39	0.40	41

Time=60 minutes; PD=25%; TU= thiourea 0.35 M;

Table 3.

Extraction of Gold by Thiourea with HCl-preleach

Time (min)	Extraction (%)			
	TU 0.525 M no preleach	TU 0.263 M with preleach	TU 0. 394 M with preleach	TU 0.525 M with preleach
15	0	4	8	10
30	6	10	22	25
45	8	16	35	41
60	11	23	44	52
75	13	28	50	60
90	16	32	59	71
105	17	34	70	81
120	19	36	72	79
135	23	33	68	78
150	26	33	62	77
165	24	32	57	76
180	22	31	52	74

TU=thiourea; HCl= 0.25 M; Temperature 25°C; PD=25%;

Table 4, the effect of Pulp Density in the same reactor.

Thiosulfate leaching results are represented by three tables: the extraction of gold in the single step process is given in Table 5; extraction of gold and silver in the double step countercurrent process is presented in Table 6, and an example of correlation between the gold extraction and the pH of the leaching solution is in Table 7.

The biological preoxidation of the mineral has a marked influence on gold and silver extraction. The addition of small quantities of an oxidant enhances the extraction by the thiourea leachant. The hydochloric preleach with additions of $Na_2S_2O_6 \cdot 2H_2O$ has a very positive effect on subsequent gold extraction by thiourea. The best results obtained were with 0.525 molar thiourea acidified with 0.25 molar HCl at 25% pulp density, after a previous HCl-sodium dithionate preleach. The temperature was maintained at 25° C.

It is very characteristic that both in case of thiourea and of thiosulfate leaching, gold extraction diminishes with time after reaching a maximum. In this study this phenomenon was almost always present. Other researchers' experiences are similar (11, 12, 20, 21), however a different interpretation of this phenomenon (22) is offered here. It is believed that the causes might be a) the high, active surface area of the solids in the pulp which causes reprecipitation of metallic gold. Consequently the pulp should be filtered after the maximum extraction is reached thus cutting the solid/liquid contact short, b) Additionally, although the double thiosulfates of gold exhibit fair stability (36), they do decompose with time. c) It is also known that in alkaline solutions gold can be instantly precipitated (37) by organic substances. Organic residues certainly are present in flotation wastes: traces of organics are able to precipitate the small amounts of gold in question, and from reaction [11] and Table 7 it can be seen that solutions become more basic with time. The diminishing gold extraction therefore, is most probably a cumulative effect of all three factors.

The fact that the solution becomes more basic is a positive development on the other hand. Not only it permits monitoring the kinetics (Table 7) of the principal reaction [11], but also protects the thiosulfate solution from spontaneous oxidation during the inproductive time and during the regeneration cycle.

Thiosulfate leaching seems to yield higher recoveries and has faster kinetics of gold extraction than thiourea leaching, similarly as reported by (20).

As mentioned, there exists a correlation between the gold extraction rate and the shape of the pH curve, as given in Table 7.

This fact can yield a lot of information about the kinetics of the process, mainly about the liberation of basic hydroxides occurring simultaneously with the gold dissolution. The highest rate of the pH change coincides more or less (there are other metals which react with the thiosulfate too) with the highest rate of gold extraction. Good S-shaped curves, however, can be obtained only if on addition of the mineral to the bath, the pH of the leachant does not fall too low, because with each pH unit down the scale, ten times as much base must be produced (or gold dissolved) to show the same effect on the pH curve.

The best results to date in thiosulfate leaching were obtained with 0.5 M leachant.

The experiments with adsorption on active carbon showed good recovery of

Table 4.

Effect of Pulp Density (%) on Gold Extraction

Time (min)	Extraction (%)		
	PD15	PD25	PD35
0	0	0	0
30	24	26	23
75	60	62	58
90	71	74	68
105	80	82	78
120	79	81	76
135	76	77	73

PD = Pulp density (%); temperature 25°C; TU = thiourea 0.525 M; HCl = 0.25 M;

Table 5.

Extraction of Gold by Thiosulfate in the Single Step Batch Process

Temp. °C	Extraction (%)								
	TS = 0.125 M FA = 0.75 M			TS = 0.25 M FA = 0.5 M			TS = 0.5 M FA = 0.0		
	30 (min)	90 (min)	180 (min)	30 (min)	90 (min)	180 (min)	30 (min)	90 (min)	180 (min)
21	10.3	12.6	26.3	50.3	48.0	43.4	59.4	48.5	60.0
37	29.7	34.3	37.7	61.7	59.4	54.3	81.7	85.7	73.7
48	31.4	37.7	31.0	44.0	43.4	44.6	78.9	88.0	90.9
63	31.4	37.1	29.1	49.7	58.3	47.3	50.3	60.0	52.0
72				16.0	48.5	36.6			

TS = $S_2O_3^{2-}$; FA = free NH_3; $(NH_3 + NH_4^+)$ = 1.0 M;PD = 25%;

Table 6.

Extraction of Gold and Silver by Thiosulfate Lixiviants in Double Step Continuous Countercurrent System

Temp. °C		Extraction (%)			
		Leach 1		Leach 2	
		ATS 0.25M + STS 0.25M	ATS 0.5M	ATS 0.25M + STS 0.25M	ATS 0.5 M
30	Au	73.1	25.7	3.4 (76.5)	26.3 (52.0)
	Ag	8.7	8.2	4.1 (12.8)	4.5 (12.7)
45	Au	63.4	90.3	11.4 (74.8)	3.0 (93.3)
	Ag	13.3	8.5	4.5 (17.8)	3.5 (12.0)
60	Au	48.0	92.6	31.4 (79.6)	7.2 (99.8)
	Ag	13.2	11.4	4.3 (17.5)	3.4 (14.8)

ATS = $(NH_4)_2S_2O_3$; STS = $Na_2S_2O_3$; M = moldm^{-3} ; PD = 11.68%; Leaching time constant = 30 min.; Values in parantheses represent the sum of extractions from Leach 1 and Leach 2.

Table 7.

Correlation between Gold Extraction and pH in Double Step Countercurrent Leaching

Time (min)	Leach 1		Leach 2	
	Extraction (%)	pH	Extraction (%)	pH
0	0.0	7.90	0.0 (86.0)	8.40
5	ND	*) 6.80	ND	8.65
10	ND	6.80	ND	8.80
15	ND	6.95	ND	8.85
20	89.0	7.10	11.0 (97.0)	8.85
25	ND	7.35	ND	8.85
30	ND	7.75	ND	8.90
35	ND	8.20	ND	8.90
40	86.0	8.35	8.0 (94.0)	8.90

*)mineral added; ND= not determined; temperature 45°C; $(NH_4)_2S_2O_3$=0.5 M; PD=11.68%; Values in parantheses represent the sum of extractions from Leach 1 and Leach 2.

of gold, but poor recovery of silver, similarly as it was observed (38) before.

Pregnant solutions have been analysed. It has been found that only traces of iron are present, that zinc dissolves only up to 3.5% of the original content in the mineral, but lead dissolves up to 43% and copper up to 36% of the original content. There is no ready explanation for this, as iron, zinc and lead are all capable of forming soluble thiosulfates, and yet they do behave differently. May be, copper and lead, being more noble behave similarly to silver and gold under these circumstances. One tenth to one fifth of the thiosulfate is being used up in each leaching step.

A two step, continuous countercurrent leaching system is suggested to improve the total yield. Table 5 reflects the results of a single step batch reactor. Table 6 and 7 for comparison, present the improved results obtained with the double, countercurrent system.

Not only higher thiosulfate ion concentrations but also higher ammonium ion content positively influence the gold extraction, as seen from Table 6.

Economic Considerations

New data on thiourea leaching (21) change the previous economic picture (33). The SKW Trostberg thiourea-ferric ion-SO_2 process is apparently able to halt the degradation of thiourea, lower the thiourea losses to negligible amounts, apply the thiourea lixiviant at much lower concentrations than known to date (e.g. (20)), and still obtain higher recoveries and better extraction kinetics. The consumption of the thiourea reagent would fall to 0.57 kg/ton permitting to extract one ounce of gold from a 0.05 oz/t mineral at the reagent cost of only $2.75, assuming $0.24 the price of a pound of thiourea. This is almost 20 times less than with 11 lb/t thiourea consumption known to date (20). Cost of reagents for cyanidation (NaCN+CaO) to extract a comparable amount of gold would be $10.50 to $42.00 depending on reagent consumption, but for thiosulfate leachant as it stands now, the cost of thiosulfate to extract an ounce of gold would reach $130.00. However thiosulfate leaching would last 1 hour, thiourea leaching 2-3 hours and cyanide leaching 24 hours minimum; sometimes weeks. Cyanide leaching also gives lower recoveries.

Although heating would be necessary to improve recovery and kinetics, the thiosulfate leachant remains potentially interesting.

Conclusions

Biological preleach and HCl-preleach proved beneficial for thiourea treatment of zinc sulfide flotation wastes.

Thiosulfate leaching however seems to give still higher results and faster kinetics, although the temperature necessary to achieve this lies around 50° C.

A double step countercurrent system of thiosulfate leaching can extract almost all gold from the mineral. Very little zinc and iron, but much lead and copper is being dissolved during the thiosulfate leaching, along with gold and silver.

The fact that the thiosulfate solution becomes more basic, has its advantages and drawbacks. Basicity permits kinetic monitoring and protects the thiosulfate solution against spontaneous oxidation; however it helps gold reprecipitation forcing a prompt separation of liquid from solids.

The results obtained in this investigation are preliminary. Several additional studies must be done to gather enough information for scaling up the processes described, in order to be able to extract gold from waste materials in an economic way.

For premined wastes, as opposed to low grade ore, there would be no mining nor crushing costs.

At this time the SKW process seems to be without competition, although SO_2 gas-handling equipment and corrosion problems might prove costly.

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