

Investigate of Effective Factors on Extraction of Silver from Tailings of Lead Flotation Plant Using Thiourea Leaching

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Abstract

In current paper, the effect of different factors on extraction of silver from tailings of lead flotation plant using thiourea leaching was studied. According to the mineralogical studies and chemical analysis taken from the tailings dam, the representative sample taken from there contained 30 ppm Ag and the minerals such as calcite, dolomite, barite, microcline, galena etc. In this research, the effects of iron(III) sulfate concentration, thiourea concentration, size and temperature onextraction of silver from Ravanj flotation plant tailings were analyzed using statistics design of experiment and DX7 software; and it was determined that iron(III) sulfate concentration and temperature were the most effective factors on the extraction of silver. The results showed that the best recovery obtained in particle size of 75 microns, 1 kg/m³ thiourea concentration and 1 kg/m³ iron(III) sulfate concentration in the temperature of 60°C for 2 hours.

Keywords

Thiourea (TU), Design Experimental (DX7), Formamidine Disulfide (FDS)

1. Introduction

Cyanidation has become the most popular method of gold and silver recycling from mineral resources because of the simplicity and economical reasons for more than a century (Wei *et al.*, [1]; Yang *et al.*, [2]-[4]). Being toxic and environmental restrictions may increase using of new reagents such as halogens (Gurung *et al.*, [5]), thiocyanide (Kholmogorov *et al.*, [6]), thiosulfate (Abbruzzes *et al.*, [7]; Ficeriová *et al.*, [8]; Hiskey and Atluri,

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[9]) and thiourea are alternatives (Çelik, [10]; Chen *et al.*, [11]; De Andrade Lima and Hodouin, [12]; Ficeriová *et al.*, [13]; Hilson and Monhemius, [14]; Kononova *et al.*, [15]; Lacoste *et al.*, [16]; Li and Miller, [17]; Muir and Aylmore, [18]; Muñoz and Miller, [19]; Murthy, [20] [21]). Some of these methods are not only eco-friendly but also their leaching rates are higher than the conventional cyanidation method (Chen *et al.*, [11]; Ficeriová *et al.*, [16]; Agma *et al.*, [22]; Groenewald, [23]).

According to aforementioned information, thiocyanide and thiourea receive much more attention than the others because thiourea has the low toxicity and kinetics higher (Wei *et al.*, [1]; Kholmogorov *et al.*, [6]; Çelik, [10]; Li and Miller, [17]; Agma *et al.*, [22]; Li and Miller, [24] [25]) but not stable and can be converted to other complex compounds easily that neutralized the gold and silver also reduces the solving rate of them; (Jinshan *et al.*, [26]; Kai *et al.*, [27]). Likewise in comparison to thiourea, thiocyanide shows less toxicity, more stability but lower solubility rate (Barbosa, [28]-[32]). Another method is thiosulfate leaching that has been analyzed by Muir and Aylmore [18], Jeffrey *et al.* [33], Feng and Deventer [34]. The disadvantage of this process is oxidation of thiosulfate and turn into polythionats that will increase the consumption of reagent.

Thiourea leaching is done in acidic medium. If ferric ion used as an oxidizing agent in the following reaction occurs in solution (Gurung *et al.*, [5]; Murthy *et al.*, [21]; Kai *et al.* [27]; Almeidaand Amarante, [35]; Gonen *et al.*, [36]; Jing-ying *et al.*, [37]; Li *et al.*, [38]; Ubaldini *et al.*, [39]):

$$Ag + 3CS(NH_2)_2 + Fe^{3+} \rightarrow Ag[Cs(NH_2)_2]^{3+} + Fe^{2+}$$
(1)

That the created complex is very strong and an oxidizing agent likes H_2O_2 or $Fe_2(SO_4)_3$ are needed to form it (Deschênes and Ghali, [40]). Formation mechanism of that complex contains two stages (TU: H_2N –CS–NH₂);

First step: a part of thiourea turns into Formamidine Disulfide (FDS) by an oxidizing agent.

Second step: FDS reacts with silver.

$$2(TU) + 2Fe^{+3} \rightarrow \frac{(NH_2)CNHSSCNH(NH_2)}{(FDS)} + 2Fe^{2+} + 2H^+$$
(2)

$$FDS + 2(TU) + 2Ag + 2H^{+} \rightarrow 2Ag(TU)_{2}^{+}$$
(3)

The total reaction number (1) is obtained from the results of two parallel reactions, number (2) and (3). Moreover, FDS can be oxidized to the unwanted products that are clear in reaction number (4).

$$FDS \rightarrow TU + H_2NCN + S$$
 (4)

In this situation, TU consumption increases and the surface of thiourea is deactivated due to the formation of the final sulfide in reaction number (4) (Arriagada and Garcia, [41]; Gönen, [42]; Marsden and House, [43]).

In the current paper, all samples are taken from the old dam of tailings of Ravanj flotation plant and it is observed that the combination of silver is in the form of silver sulfide or argentite and it is about 30 ppm.

2. Materials and Methods

2.1. Sample Characterization

The samples taken from wells are drilled by powder drilling machine (RC 100) in tailings damp are mixed and the representative sample is provided for analysis. Sieve analysis and mineralogical studies of these samples are illustrated in Table 1, Table 2 and Figure 1.

As it is shown in the table, calcite and dolomite have the highest percentage of minerals in this sample which shows the carbonate nature of tailing ore. These studies showed silver is lucked with galena and the liberation degree of silver was about 53 microns.

2.2. Preparation and Experimental Procedure

Experiments were conducted in three dimensions, so that the samples were crushed by Laboratory wet rod mill (D = 16 cm, L = 35 cm) for 15, 20 and 25 minutes. The results are shown in Table 3.

The crush sample was divided into 500-g-smaller samples. All tests were done in Ravanj mine laboratory with its complete equipment. To reduce the pH, thiourea leaching should be done in acidic medium so 30%-sulfuric acid is used. At first, because the highest percentage of minerals in samples contain carbonate minerals so a heating stage up to 500°C was performed in furnace before leaching process (Laboratory furnace 2.5 lit).

Table 1. Sieve analysis of representative sample from tailings of Ravanj flotation plant.								
Fraction of Dimensions	Fraction of Dimensions Weight		Cumulative weight oversize	Cumulative weight undersize	Grade	Distribution		
(μm)	(gram)	- Percentage (%) -	(%)	(%)	(%)	(%)		
212	11.9	6.24	6.24	93.76	15.5	5.2		
-87	21.6	11.32	17.56	82.44	17.7	10.8		
-50	27.8	14.57	32.13	67.87	19.8	15.6		
-22	24	12.58	44.71	55.29	20.6	14		
-53	105.5	55.29	100	0	18.2	54.4		
total	190.8	100						

Table 1. Sieve analysis of representative sample from tailings of Ravanj flotation plant.

 Table 2. Mineralogical composition of representative sample from tailings of Ravanj flotation plant using XRD method.

Minerals	Formula	Percent	
Calcite	CaCO ₃	32.2	
Dolomite	CaMg(CO ₃) ₂ /CaO·MgO·2CO ₂	19.8	
Barite	$BaSO_4$	10.2	
Quartz	SiO_2	12.3	
Sphalerite	ZnS	4.4	
Galena	PbS	3.7	
Microcline	KAlSi ₃ O ₈	11.3	
Illite-2 ITM2 RG	$K_{0.7}Al_{2.1}(Si,Al)_4O_{10}(OH)_2$	6.1	

Table 3. Grinding time of sample from tailings of Ravanj flotation plant.

Grinding time (minute)	Particle diameter		
(minute)	$d80 = \mu m$		
15	75		
20	53		
25	45		



Figure 1. Mineralogical analysis with thin section.

Main experiments were done in a beaker (1000 mL) with 40%-solid concentration and constant speed stirrer (200 rpm) on a hot plate with an adjustable temperature. To prevent the possible changes in pulp density occurrence (because of the solution evaporate) distilled water was added to pulp during the experiments. The pulp was mixed with sulfuric acid (30%) to reach pH 1. The time needed for each test was at least 2 hours. Silver extracted from solution was analyzed by the atomic absorption spectrophotometry (Thermo model iCE 3300GF) in high temperature. The extraction of Ag was also calculated by using grades and leach solution volume and residue leach weight resulted from the experiments after decomposing the TU complex with HNO₃ at high temperature.

2.3. Design of Experiments

In order to evaluate effect of variables and their interaction DX7 software and statistical design of the experiment were used. The advantages of designing such statistical experiments are lower related cost and reduced number of experiments (Fatahi *et al.*, [44]; John Wiley & Sons [45]). In this research incomplete factorial designing is recruited with regard to relation $N = 2^{n-1}$ (N = number of tests, n = number of variables). These variables such as TU concentration and iron(III) sulfate concentration, temperature and dimension were checked in three levels (low, middle and high). Table 4 shows the type and value of these factors. Table 4 shows the type and value of the variable.

3. Results and Discussion

Leaching tests were designed and done based on four variables and three repeating experiments as center point. The **Table 5** shows the conditions and results of design in DX7.

The results show that the maximum recovery obtained in test number 5 in 80.8%. The following equation is obtained from a mathematical model that can predict the amount of silver recovery by putting different variables (Equation (5)). In this model, the recovery percentage is predicted considering the effective variables¹:

$$Recovery = +60.40000 - 8.08750B + 0.46000C$$
(5)

In the above equation $B = Fe_2(SO_4)_3$ and $C = Temperature^2$. By using ANOVA analysis, the effective factors are determined which are available in Table 6.

If the F-value of the variable is higher than F-value of the model, so it shows the high effect of that variable with confidence percentage of 95%. The results show that iron(III) sulfate concentration and temperature are effective on Ag recovery. The most effective factor on Ag recovery is iron(III) sulfate concentration. **Figure 2** shows the normal plot curve which confirmed statistical analysis.

The effect of iron(III) sulfate and temperature is shown in **Figure 3** and **Figure 4**. As can be seen in the figure, increasing the iron(III) sulfate concentration from 1 to 3 kg/m^3 leads to reduction of recovery from 73% to 57%. This reduction is due to the combination of some iron(III) sulfate with thiourea and reduction of thiourea concentration consequently. Solve this problem by adding new thiourea or reduce iron(III) sulfate concentration is possible. It seems optimizing iron(III) sulfate concentration to avoid increasing the cost more reasonable³.

Also Increasing temperature from 30° C to 60° C increased the silver recovery rate. Higher temperatures speed up decomposition of thiourea and if the main variables such as the concentration of Iron(III) sulfate and thiourea be appropriate thiourea will form a complex with silver normally.

variable	symbol	Unit –	Coded variable level		
			Low	Center	High
Thiourea	А	Kg/M ³	1	3	5
$Fe_2(SO_4)_3$	В	Kg/M ³	1	2	3
Temperature	С	°C	30	45	60
Size	D	μm	45	60	75

Table 4. Coded and actual levels of independent variables used in factorial design.

¹Obtained DX7 software.

³Refer to Introduction, Equation (2).

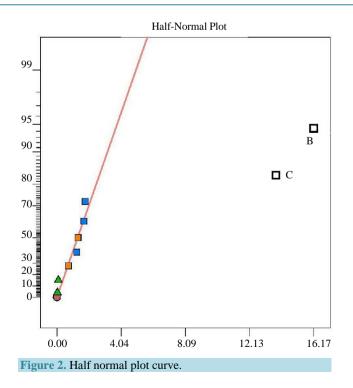
²Table 4.

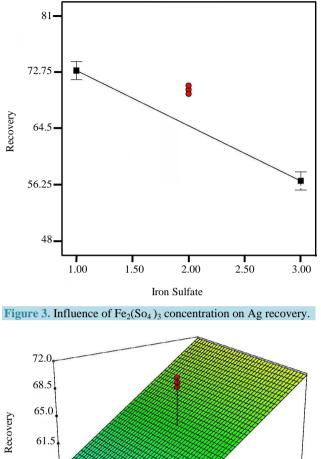
Table 5. Factorial design and experimental results infourea leacning experiments.									
Dam Ma	Coded level of variables				Actual level of variables				Response
Run No	А	В	С	D	А	В	С	D	(%)
1	-1	-1	-1	-1	1	1	30	45	68.25
2	1	-1	-1	1	5	1	30	75	64.7
3	-1	1	-1	1	1	3	30	75	48.25
4	1	1	-1	-1	5	3	30	45	50.9
5	-1	-1	1	1	1	1	60	75	80.8
6	1	-1	1	-1	5	1	60	45	78.3
7	-1	1	1	-1	1	3	60	45	65.8
8	1	1	1	1	5	3	60	75	62.4
9	0	0	0	0	3	2	45	60	70.2
10	0	0	0	0	3	2	45	60	69.6
11	0	0	0	0	3	2	45	60	70.8

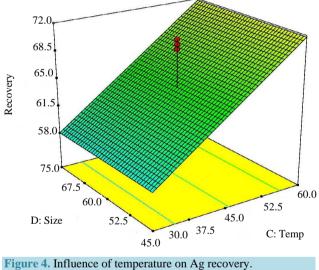
Table 5. Factorial design and experimental results thiourea leaching experiments

Table 6. Results obtained from the ANOVA analysis.

Source	Sum of squares	Df	Mean square	F-value	p-value prob > F	Note
Model	904.14	2	452.07	154.45	< 0.0001	significant
$B-Fe_2(So_4)_3$	523.26	1	523.26	178.77	< 0.0001	
C-temp	380.88	1	380.88	130.13	< 0.0001	
Curvature	60.71	1	60.71	20.74	0.0026	significant
Residual	20.49	7	2.93			
Lack of Fit	19.77	5	3.95	10.98	0.0856	not significant
Pure Error	0.72	2	0.36			
Cor Total	985.34	10				







4. Conclusion

Mineralogical studies of representative sample from tailings of Ravanj flotation plant indicated silver was lucked with galena and showed that it contained 30 ppm silver and the highest percentage of minerals in it was carbonate minerals that increased the consumption of sulfuric acid. So acidic leaching would not be economical in these samples. For this reason, a heating stage up to 500°C was performed before main tests. Modeling and optimization, results analysis and experiments design process were done by DX7 software. ANOVA results indicated that temperature increase and Iron(III) sulfate concentration decrease were the effective factors on the silver recovery. Maximum recovery of silver occurred in size of 75 microns with 1 kg/m³ thiourea and 1 kg/m³ iron(III) sulfate in 60°C for 2 hours.

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