Recovering Lead Metal from Lak Mine Lead Concentrate by Ferric Chloride Leaching

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Lead concentrate from Lak lead mine in Iran was processed via leaching method. In this method some of the undesirable conditions such as long term melting and roasting and sulfur gas emission prevailing in the conventional pyrometallurgical lead extraction method are omitted. Therefore, it is more environmentally friendly to produce lead by leaching method. The use of ferric chloride and sodium chloride as leaching reagents at various temperatures was examined. It has been shown that 99.8% lead and 82.2% silver can be extracted from sulfide concentrates. According to this research, the optimum conditions for Lak mine concentrate are: Ferric Chloride: 45gm/lit, Sodium Chloride: 200gm/lit. temperature: 70°C and the leaching time: between 9 to 12 minutes.

Keywords: Leaching method, Sulfur emission, Lead concentrate.

1. INTRODUCTION

Many lead ore deposits which contain silver are generated within the earth by mean of warm hydrothermal fluids. These fluids may be trapped below the surface in cracks where galena containing silver and other minerals may precipitate to make vein deposits. Where limestone occurs the fluids may fill cavities to form rich but patchy lead-zinc-silver deposits. Lead ores often contain significant amounts of silver [1].

The run of mine ore is ground and passed through various processing stages including gravity separation and froth flotation [2]. The product of flotation stage is a concentrate, which may be originated from sulfide or oxide ores. In order to extract the lead content of the concentrate further refinement need to be done. Pyrometallurgy is an old method which is no longer desired by metal extraction industry because the flue gases should be controlled, and also it requires environmental procedures. This econo-environmental factor associated with the sintering and smelting process along with the concern over acid rain, which will result in even more stringent controls on emission of sulfur gases caused lead producers to pursue alternative methods.

A method for the production of Pb metal from galena (PbS) concentrates was developed and introduced as an effective low-temperature leaching-electrowinning method [3,4]. The method reduces Pb emissions and totally eliminates the formation of sulfur gases. The elemental sulfur produced is more economical to store and ship than that of the sulfuric acid (H_2S0_4) generated by the high-temperature smelting process. This hydrometallurgical method of lead extraction receives much appreciation today. In the leaching method, some of the undesirable conditions such as long term melting and

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roasting and sulfur gas emission are omitted. Therefore, it is more environmentally friendly to produce lead by leaching method. Several galena leaching processes have been investigated, including processes using ferric chloride [5], ferric sulfate [6], and nitric acid [7]. Chloride leaching, among other processing techniques, is the most recognized and widely used recovery method. Chloride leaching processes have been employed using either NaCl or $MgCl_2$, and $CaCl_2$ or $FeCl_3$ [8-14].

Figure 1 graphically shows the method used by some researchers like Gupta and Mukherjee [15] for treating galena concentrate. Here, the leaching of the ground concentrate is done with a lixivant composed of $FeCl_3$, NaCl and $PbCl_2$ at 100°C for only about 15 mins. Then, cooling of the leach liquor is done to crystalize $PbCl_2$. As can be seen, a product of pure lead and sulfur higher than 99.85% is obtained and leach liquor is also regenerated.

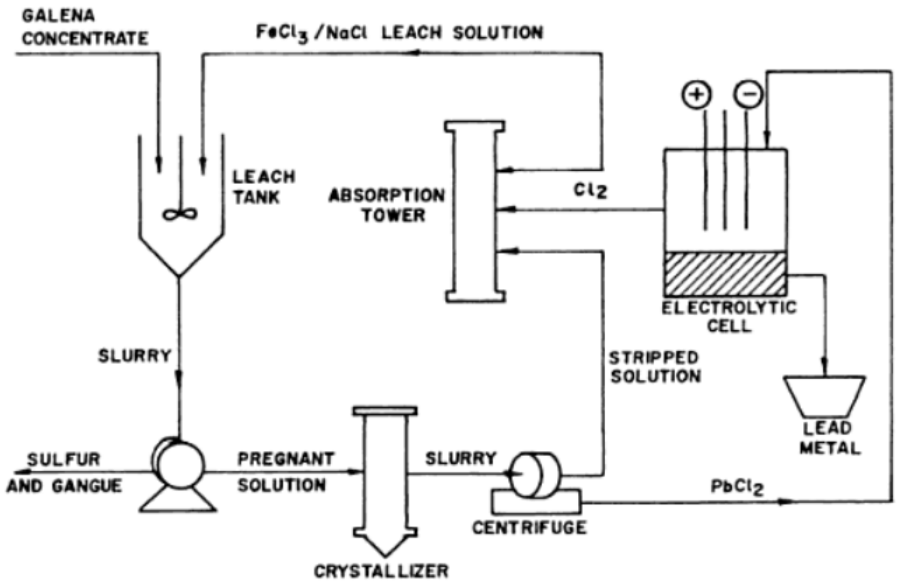

Fig. 1: Flow diagram for recovering lead by $FeCl_3$ leaching.

The aim of the investigation is to demonstrate the effectiveness of the use of ferric chloride along with sodium chloride as leaching reagents in extracting lead and silver metals from lead concentrate. Some important hints about the effects of NaCl on the enhancement of ferric chloride activity will be given. The role of temperature on recovery of lead and silver in chloride leaching of lead concentrate will be examined.

2. MATERIALS AND METHODS

Commercial grade common salt (NaCl), as a cheap agent was used to prepare chloride solution. Ferric chloride, standard grade was provided by Sitara Chemical Industries Limited. Ice cubes were also prepared as required. Other equipment includes glass containers (1.5 liter capacity), a thermostatitic bath, a digital balance, sampling tools, and a hot plate lab stirrer with controllable temperature.

3. EXPERIMENTAL PROCEDURE

The Lak lead and silver ore mine is located in Qazvin, Iran. Geologically, a number of massive diorite and granodiorite resulted in lead mineralization in the region. There exist well mineralized sections across a famous fault called Boin Zahra. Lead concentrate is produced in a small mineral processing plant nearby. Galena concentrate samples were obtained weighing 02 kg in dry powder (finer than 75 microns in size). The lead and silver content was obtained via atomic adsorption spectrometer: Pb: 58.5% and Ag: 961ppm. Table 1 shows an elemental analysis of the concentrate sample. The lead concentrate offered by the world market typically contains the same percentage of lead, but the proportion of silver is quite lower (around 10 time) than that of the Lak mine [16]. Table 1 shows an elemental analysis of the concentrate.

Table 1: Elemental analysis of the lead concentrate.

Element	**%**
Pb	58.491
S	15.522
Fe	12.903
Mg	4.391
Ag	0.0961
Ca	2.302
Sb	0.153
Ca	6.154

The experimental procedure for all tests was as follow:

a) preparing 500ml solution of ferric chloride ($FeCl_3$) powder in deionized water by stirring the solution (200gm/lit)

b) adding 100gm lead concentrate powder to this solution after leaving it in a bath with increasing temp.

c) adding sodium chloride to the solution while controlling the time at the desired level.

Samples were taken from the solutions before being filtered. The experiments solutions contain sodium chloride, silver chloride, ferric chloride and lead chloride.

The above step by step procedure was repeated for each test. Table 2 shows the experimental design parameters and levels chosen, and Table 3 shows the settings numbered for all tests. As can be seen nine key tests were conducted.

Table 2: The experimental design parameters.

Parameters	**Level 1**	**Level 2**	**Level 3**
Temperature (°C)	50	70	80
NaCl (gm/l)	0	100	200
$FeCl_3$ (gm/l)	35	45	50

Table 3: the settings of parameters for the nine key tests

$FeCl_3$ (gm/l)	**NaCl (gm/l)**	**Temperature (°C)**	**Test No**
45	200	50	1
45	200	70	2
45	200	80	3
45	0	70	4
45	100	70	5
45	200	70	6
35	200	70	7
45	200	70	8
50	200	70	9

As can be seen in Table 3, in tests No 1 to 3 the temperature was changed between 50°C to 80°C whilst keeping the other two parameters constant. Similarly, in tests No 4 to 6, NaCl concentration was altered from 0 to 200gm/l, and in tests 7 to 9 $FeCl_3$ concentration was changed from 35 to 50gm/l. For all tests, the heating conditions were under control while stirring the solution in the bath. Followings are the continuation of the testing procedure (stages a-c):

d) in order to let lead and silver chloride crystals to precipitate, the solution was cooled by ice cubes from 70°C to 10°C.

e) the solution was filtered again to separate lead chloride.

f) the lead chloride crystals were dissolved in sodium chloride solution once more for separating pure lead.

This final stage solution is an electrolyte from which pure lead (Pb: 99.98%) can be extracted. The other part, silver sludge is also a source of silver, which can be extracted via compilation melting. Also, the extra ferric chloride ($FeCl_3$) and sodium chloride (NaCl) can be recycled for use in the subsequent experiments as shown in Figure 1.

It is noteworthy to mention that while mixing in the solution in the thermostatitic bath (stage c) the temperature was kept at the desired level, and as accurate as possible. In order to study the kinetics of the leaching process the sampling was done at interval times (6, 9, 12 and 15 mins). In total, 36 samples were taken. After placing the solution in the ice container the lead and silver chlorides precipitation was noticed; then, the solution was filtered. Samples were sent for analysis using an atomic absorption spectroscopy for lead and silver content.

4. RESULTS AND DISCUSSIONS

The results of the sample analysis were shown in Table 4. As can be seen, by increasing temperature from 50°C to 70°C, the recovery of Pb and Ag is considerably increased (the recovery of Pb rises from 93.46% to 99.06%, and the recovery of Ag increases from 75.6% to 82.18%, but further increase in temperature to 80°C causes a decline in recovery. Thus, 70°C is considered as the optimum point. In the second stage both $FeCl_3$ concentration and temperature were kept constant while NaCl concentration was changed. As can be seen in Table 4, by increasing NaCl concentration the recovery of Pb and Ag is increased. But the increase is more pronounced for Ag than for Pb. Thus, 200gm/l for NaCl is considered as the optimum point. In the same way, an optimum point of the order of 45gm/l was found for $FeCl_3$ concentration.

Table 4: The analysis results for 36 samples taken from 9 key tests.

Time	**6 mins.**		**9 mins.**		**12 mins.**		**15 mins.**	
Test No.	**Pb%**	**Ag%**	**Pb%**	**Ag%**	**Pb%**	**Ag%**	**Pb%**	**Ag%**
1	82.6	68	92.1	74.2	93.2	75.2	93.46	75.6
2	94.01	75.9	99.2	82.25	99.12	82.42	99.19	82.68
3	93.25	76.1	98.25	82.3	98.79	82.26	99.06	82.18
4	89	15	92	23	92.7	25	93	25.5
5	92	62	97.2	68.3	97.5	68.9	98.2	69.1
6	93.6	76.3	98.9	82	99.1	82.8	99.2	82.9
7	92.4	75.1	98.7	81.89	98.85	81.92	98.89	82.01
8	93.4	76.5	99.13	82.85	99.15	82.9	99.3	83.1
9	92.9	74.9	98.6	81.95	98.12	81.97	98.9	81.97

Gupta and Mukheerjee [15] pointed out that galena can be easily dissolved (oxidized) in $FeCl_3$ solution according to the following formula:

$$PbS + 2\,FeCl_3 = PbCl_2 + 2\,FeCl_2 + S$$

They mentioned that combination of $FeCl_3$ leaching and fused salt electrolysis is more efficient. This is also practiced in this study. The lixiviant composed of $FeCl_3$/NaCl is kinetically fast as indicated in Table 4. However, they estimated that 2,5 lit of $PbCl_2$-NaCl solution is needed to produce 182gm $PbCl_2$ from 200gm of concentrate. This high amount of mixed solution to dissolve the $PbCl_2$ formed in the leaching seems to be too much requiring large size cells. This implies a high cost, but the use of relatively low residence time multiple medium size cells is likely to be an alternative feasible way.

5. CONCLUSIONS

This study suggests the hydrometallurgy method for lead extraction. It has been shown that 99.8% lead and 82.2% silver can be extracted from sulfides concentrate. However, further investigations would be useful for overcoming the problem of loss of some proportion of silver. According to this research, the optimum conditions for lak mine concentrate are: Ferric Chloride: 45gm/lit, Sodium Chloride: 200gm/lit., and temperature: 70°C. These are the conditions which could indicate a better result over pyrometallurgy.

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