JME

***J**ournal of **M**ining & **E**nvironment,*
Vol.3, No.2, 2012, 79-87.

Implementation of flotation and gravity separation, to process Changarzeh sulfide-oxide lead ore

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Received 25 Sep 2011; received in revised form 1 Feb 2012; accepted 16 May 2012
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Abstract

This study aimed to explore concentration of a low grade sulfide-oxide lead-zinc sample containing 2.3% Pb, 1.91% Zn taken from Changarzeh mine, South Natanz, Iran. The effects of different parameters such as type and dosage of collector, milling retention time, dosage of sodium sulfur and its preparation time, application of sodium silicate, pH and solid content were investigated in relation to flotation efficiencies. Optimum experiment was carried out in cumulative flotation with 200g/t KAX as collector, 2000g/t Na_2S, 500g/t sodium silicate, 30g/t MIBC and at pH=10. This test yielded 94% of lead recovery under optimum condition performance, and a concentrate with 70%Pb was produced through cleaning stages. Eighteen minutes was found to be the optimum time for lead flotation in laboratory scale. Application of gravity method for the production of a middle product was also considered. For shaking table the effect of table slope and water flowrate and for jig the effect of water flowrate and frequency were studied. Gravity separation by shaking table resulted in a concentrate with 46%Pb and 80% recovery, so shaking table could be proposed for production of pre-concentrate.

Keywords: *Oxide ore; Cerrusite; Flotaion reagents; Gravity Concentration.*

1. Introduction

The lead oxide minerals are cerrusite, anglesite and plambojarusite and the common method for the recovery of these minerals is flotation [6]. Oxidation degree in lead ores varies from tiny tarnish to complete oxidation [7]. Nowadays effective beneficiation of the oxide-sulfide ores is a great concern for engineers. Low floatability of these minerals is a result of their high solubility [3]. Lead ions solved from the mineral structure react with collector and perticipitate as the insoluble lead xanthate and increase the consumption of collector [2]. It was found out that through cerrusite flotation, collector consumption is much higher than the required amount for galena flotation [2, 4]. These researchers declared that the main reason for high consumption of collector is most probably the reaction between X^+ and $PbCO_3$, where X^+ could be Pb^{2+}, Cu^{2+} and/or Zn^{2+}. As a result, the oxide lead minerals are treated by sulfidizing agents before being floated [2, 3, 4]. Adding sulfide salts to the system sulfidizes the oxide mineral surface and makes it hydrophobic, to be floated easily. As a result, collector consumption decreases [2].

When the oxide lead minerals exist in considerable amounts, first the lead and zinc sulfides and then the treated oxide minerals are floated. The amount of sulfidizing agent for different ore types is different and normally between 500 to 2500g/t. Precise control is needed in sulfide addition, because while the shortage of sulfide ions will cause ineffective sulfidation, over consumption of sulfides will depress sulfide and sulfidized minerals [3]. Aero 404, 407 or 412 in combination with isopropyl or amyl xanthate, such as sodium isopropyl xanthate (Z11) and potassium amyl xanthate (KAX), are the preferred collectors for flotation of lead minerals. Adding sulfides and collectors in stages is a common method in rougher flotation [7]. Also flotation of oxide-sulfide minerals by xanthates is strongly dependant on the pulp pH. Lead recovery decreases by a pH over 10 [8, 10]. Primary studies on semi oxide flotation ores have declared that flotation of these ores is more complex than that of oxide ores. It needs the mix of different collectors and high consumption of chemicals such as sodium sulfide. Although there is a general knowledge regarding the flotation characteristics of these ores, each ore should be considered uniquely according to its mineralogical characteristics.

Flotation is recognized as the conventional approach to process the sulfide-oxide minerals. There exist other methods for the process optimization; among them are the physical separation methods, like gravity concentration which is used for concentrate and preconcentrate production [7]. Using gravity methods prior to flotation is an effective method to increase the flotation feed grade, and also tail removal for decreasing the amount of feed entering the milling and flotation process; this leads to an economical save. Weak response of sulfide-oxide ores to flotation, because of high solubility of these ores and as a result of collector high consumption, has given a very important role to the gravity methods [9].

If liberation of lead particles is achieved at coarse sizes (over 500 microns), the gravity methods could be employed because under this condition the specific gravity of lead minerals will be higher than that of the tail. For finer particles, flotation should be used.

In a number of references it is declared that gravity methods are not used extensively. Even when a floating low grade tail is produced by gravity separation, it is possible that by liberation of valuable particles, these particles could also be recovered. Generally, in each plant, the economic concerns define the methods [11].

On the other hand, gravity methods have considerable advantages, including low installation cost per ton, low energy cost such as comminution costs, high capacity, effectiveness and lack of harmful environmental effects in comparison with other methods like flotation because of not using the organic chemicals during separation process [5].

This paper presents the results of the reseach work conducted for obtaining the maximum recovery of the oxide-sulfide lead minerals from Changarze ore.

2. Materials and methods

The sample was provided from the Changarze mine, Natanz, Iran, by estimated weight of about 90kg and the original size of 10-15cm. The sample was from about 15 points of a heap, so as to cover all the the heap surface. About 4-5kg of sample was taken from each pointfrom the surface and a hole with depth of 0.5 meter in all 15 points. Mineralogical studies revealed that primary minerals of Changarze ore were galena, copper minerals and pyrite, and secondary minerals were cerrusite, coveline, malachite, azurite and wulfonite. Gangue minerals were calcite, quartz and barite. The XRD analyses on the sample showed that dolomite, quartz, ankerit, gismondit, galena, cerrusite, tetrahedrite, anglesite, pyrite, chalcopyrite, sphalerite, calcocite, calcite, albite and gypsum were the main constituting minerals of this ore respectively (Figure 1). Also existence of galena and cerrusite as lead minerals, and coveline, pyrite, tetrahedrite, dolomite and quartz as gangue was also confirmed. Feed studies by the electronic microscope showed that more than 50% of galena altered to cerrusite as a result of oxidation. Lead grade was reported as 2.3% and zinc grade as 1.98% through feed analyses by atomic absorption method. Studies of mineral liberation revealed that at the size of 212 microns, 95% of the particles of galena accompanied by cerrusite were liberated and for galena itself, 85-90% of particles were released at this size.

To achieve the desired size, first the sample was crushed to d_{80}=2360 microns. For gravity operation this size is sufficient. To prepare the flotation samples, the product underwent a milling process for 10 minutes, so as to liberate the valuable particles at the size of 212 microns.

Flotation tests were carried out in a 2-liter cell, using Denver flotation machine of 800rpm and the solid content of 37% in weight. In these experiments, potassium amyl xanthate was implemented as collector, sodium sulfide as activator for the oxide minerals, MIBC as frother, and sodium silicate as dispersant. Table 1 presents the chemicals used for the primary experiments.

Through flotation, the effect of qualitative parameters such as simultaneous or separate recovery of sulfide and oxide lead minerals collector and frother type were studied. Also the optimum amount of quantitative parameters like the mill retention time, collector, sodium sulfide and pH for the maximum recovery of lead were determined. Then the final tests for cleaning of rougher concentrate and increasing its grade were performed.

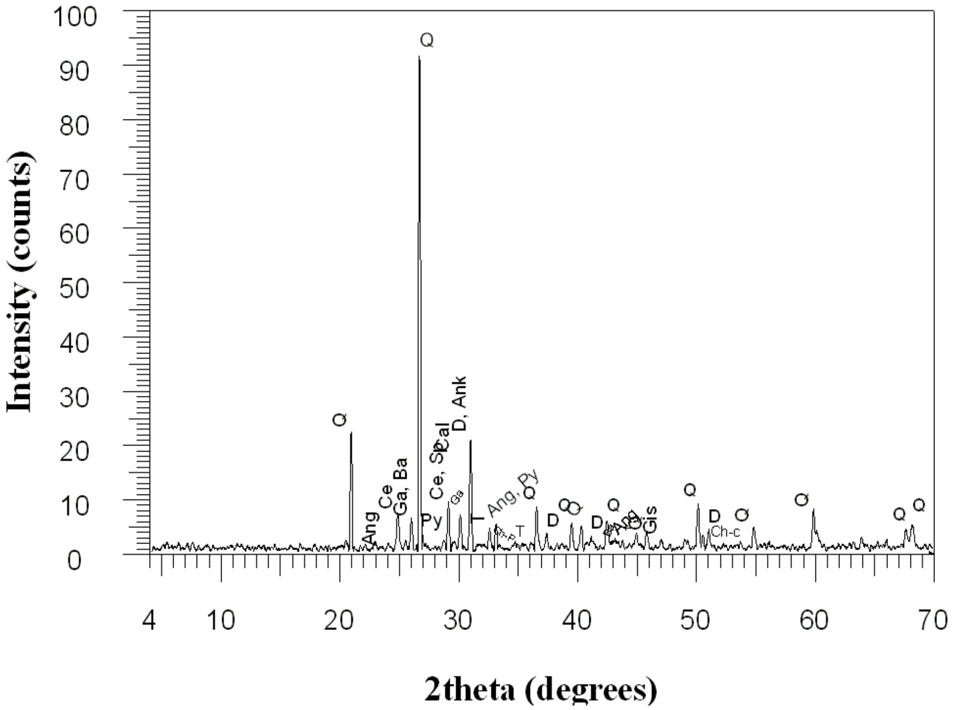


Figure 1. The XRD graph of the sample.

Table1. The chemical reagents used in the flotation experiments

Chemical reagents	Dosage (g/t)
NaOH	360
Z11	200
KAX	200
Na_2S	2000
pine oil	20-60 (1-3 drops)
MIBC	20-60 (1-3 drops)
Aeroflot 65	20 (1 drop)
pH	8.4-10.4
$NaSiO_3$	500
H_2SO_4	750

3. Results and discussion

3.1. Flotation

Different experiments were performed to determine the optimum amount of parameters. Effect of each change of parameter on recovery in rougher stage was surveyed as follows.

3.1.1. Effect of cumulative and sequential flotation

To investigate the effect of sequential flotation, lead sulfide minerals were floated first and then, after activation of oxide minerals, they were also recovered. Sulfidation process for cumulative flotation was carried out at the beginning, and sulfide and oxide minerals were recovered simultaneously. Lead recovery in concentrate gained at sequential flotation was 87.27% which is lower than that of cumulative flotation, 88.99% (Figure 2). Sequential flotation demonstrates good results when lead oxide minerals are present in considerable amounts [7].

3.1.2. Collector type

Xanthates are the most common collectors in flotation of oxide and sulfide lead ores [10]. Comparing isopropyle and amyl xanthates, the undesirable effect of isopropyl xanthate on lead recovery is apparent. Using the same amount of Sodium Isopropyl Xanthate (Z11) instead of Potassium Amyl Xanthate (KAX) decreased lead recovery from 96.03% to 92.83% (Figure 3).

Isopropyl potassium xanthate is a long chain collector wile potassium amyl xanthate is a short one. The longer the collector chain, the lesser is the needed collector concentration for its salt precipitation. Along the same lines, the longer the collector chain, the more hydrophobic is the mineral surface [9]. With 200g/t collector concentration, it is predictable that longer chain collector has better effect on making surface more hydrophobic.

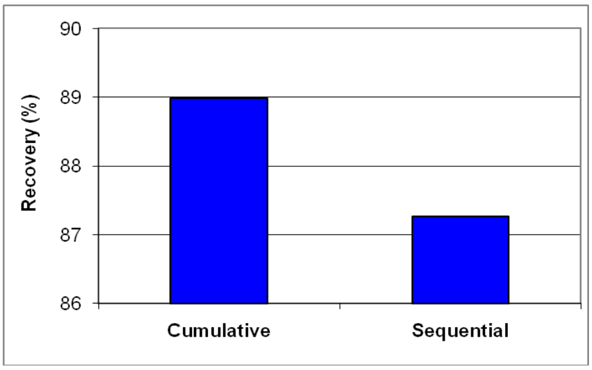


Figure 2. Lead recovery for cumulative and sequential flotation.

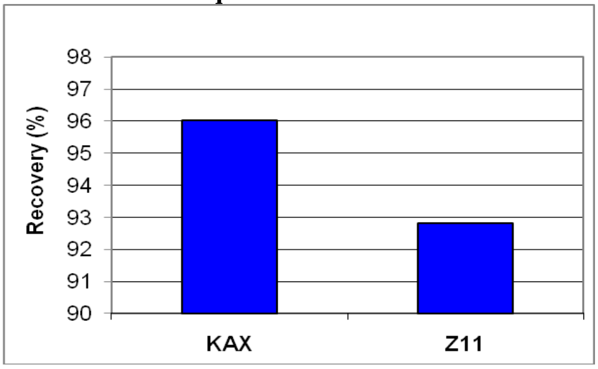


Figure 3. Effect of collector type on lead recovery.

3.1.3. Effect of increasing the collector dosage

On recovery curve of lead versus xanthate dosage (Figure 4), recovery increases to a maximum point by increasing xanthate concentration and at a certain point, it remains constant. If KAX is used for galena, 220-315g/t gives the best recovery [2]. To determine optimum dosage of collector for Changarze ore, 200g/t KAX in basic test was increased to 300g/t, but a favorable change was not observed in concentrate lead recovery (Figure 4). Also since the 200g/t of collector was added in two stages of 150 and 50g/t, and the added 50g/t was effective on floating load, 200g/t of amyl xanthate is considered necessary.

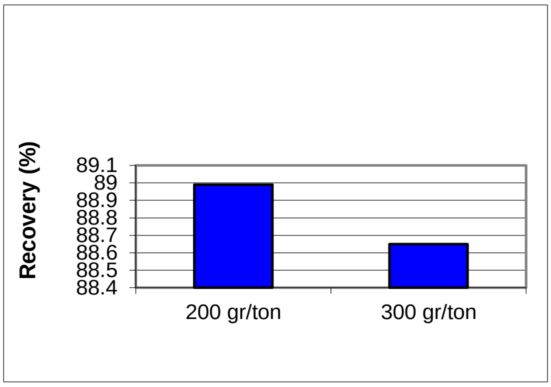


Figure 4. Effect of collector dosage on lead recovery.

3.1.4. Effect of added sulfidizing agent

Desirable effect of sulfidation of oxide minerals is known from the early days of flotation [2]. After sulfidation process, surface will become less hydrophilic in presence of chemisorbed sulfide ions and will float more efficiently. Under this condition, collector consumption will decrease considerably. When sulfur consumption exceeds $5.3*10^{-4}$ mol/liter, depression will occur as a result of sulfur absorption instead of xanthate [9].

In these series of experiments, the amount of sodium sulfur was changed between 1500-3000g/t. Increase in sodium sulfide dosage led to an increase in the lead recovery in concentrate from 83.72 to 88.99%, and then the recovery decreased to 86.87% by increasing sodium sulfur to 3000g/t. Therefore, the 2000g/t was accordingly determined as the optimum amount of sulfidizing agent (Figure 5).

3.1.5. Effect of sodium sulfide preparation time

To determine the optimum time for sulfidation of oxide minerals, the preparation times of 3, 6 and 10 minutes were studied. The results revealed that the 6 minutes preparation time resulted to the optimum recovery of 96.03%. Probably the long sodium sulfide preparation time would depress galena like its huge consumption.

A wide range of 30 seconds to 15 minutes is recommended as preparation time of sodium sulfide. It has been reported that the sulfidation time is 30 seconds [2].

3.1.6. Adding collector and sufidizing agent in stages

Adding chemical agents in stages prevents sudden increase of pH which leads to a decrease in electric potential and a resultant decrease of Eh and depression of lead minerals [3, 4].

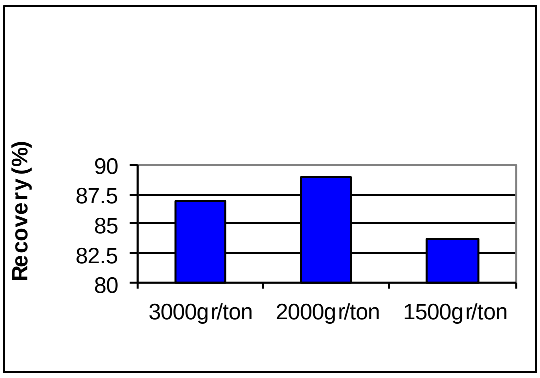


Figure 5. Effect of Sodium Sulfur dosage on lead recovery

The effect of adding collector and sulfidizing agent in stages was studied. Collector and sodium sulfide were added to the pulp in five stages. Despite what was expected, the results were not desirable compared to the base experiment; the recovery of staged experiments decreased to 77.76% (Figure 6). Since this process was a complex and time-consuming one, it was not considered in the following experiments.

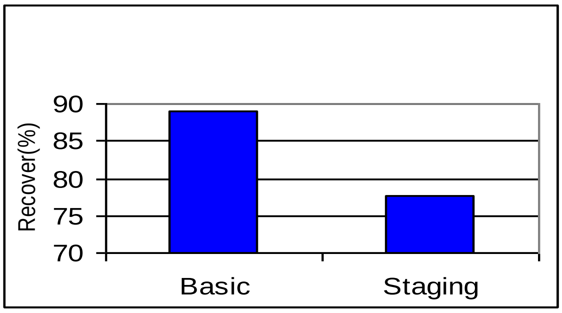


Figure 6. Effect of multi stage addition of chemical on lead recovery.

3.1.7. Effect of sodium silicate

Fine particle production during grinding process increases the specific surface of particles and chemical consumption. These particles cover the particle surface and lead the valuable minerals to tail. On the other hand, since a great proportion of the valuable minerals exists in the form of fines, fine removal will not be an effective method to be applied. As a result, in another experiment the effect of sodium silicate addition as dispersant of fine particles was studied. In flotation of some minerals, the agglomeration of fine particles will create problems in flotation control and causes a decrease in recovery and grade of concentrate. Thus, when the amount of these coarser particles is high, the sodium silicate is used as dispersant [9].

As the results show, addition of sodium silicate had a very desirable effect and increased the lead recovery in concentrate from 66.66 to 74.82%. Consumption of sodium silicate is very sensitive in such a way that a definite amount of it leads to a desirable result, but an increase or decrease in its consumption leads to undesirable results [9]. In this test by increasing the amount of sodium silicate from 500 to 750g/t, recovery decreased from 74.82 to 66.66% (Figure 7).

3.1.8. Effect of pH control

Complete flotation of galena was carried out through pH=2-10. Over the pH=11, as a result of production and stability of lead hydroxyl (polombit), galena will be depressed. This depression is due to the production of plombit in place of lead xanthate on the galena surface [9]. Also at high pH, iron hydroxide which is probably entered from steel mill or the pyrite content of the ore, to the pulp, will produce the hydrophilic metallic oxi-hydroxi and convert the surface to a hydrophilic surface, and floatability will decrease [1]. By adjusting pH at 8.5-9 criteria, lead recovery in concentrate will increase from 88.99 in the base test to 91.28% (Figure 8).

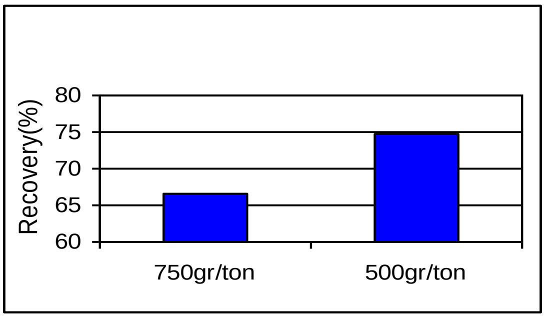


Figure 7. Effect of Sodium Silicate dosage on lead recovery.

3.1.9. Simultaneous effect of low pH and sodium silicate

To study the simultaneous effect of low pH and sodium silicate, an experiment was performed. The results show that the simultaneous increase of these two factors decreased the recovery to 74.82%. Since each of these two factors increase the recovery, selection of one of them is necessary for attaining optimum pH. The interactive effect of these two factors can be studied through the design of experiments.

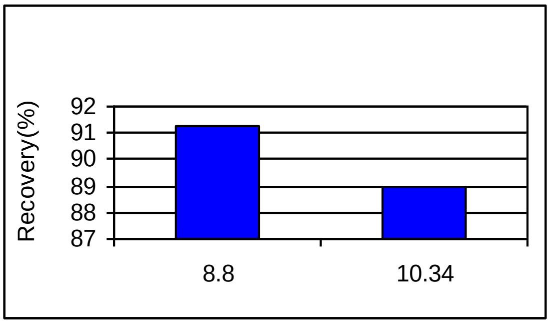


Figure 8. Effect of pH on lead recovery.

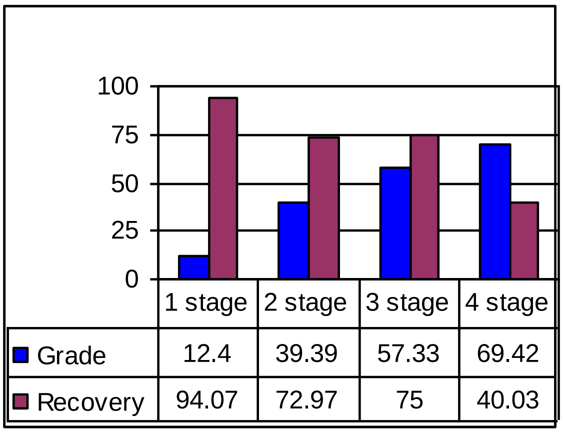
3.1.10. Effect of frother type

To select the frother which is more compatible to the test circumstances, MIBC, pine oil and Aerofloat 65 were tested. The best result was gained by MIBC (recovery of 94.07%). The recovery of pine oil was 90.48%. To improve froth condition and increase the froth depth, Aerofloat 65 with high frothing characteristics was used. Although a stable and deep froth was produced, the recovery decreased to 72.23%.

Certainly frother type is different in various conditions. While the combination of low pH and sodium silicate was being studied, it was observed that MIBC was not effective in this condition. At the same condition, adding pine oil improved the froth condition.

3.1.11. Effect of the number of cleaning stages

In the final stage, increasing the concentrate grade by employing the minimum cleaning stages (with no chemical addition) was studied, when the optimum conditions of rougher flotation was determined. The results of the experiments are shown in Figure 9. By increasing the number of cleaning stages, the lead grade in concentrate will increase. Finally in the fourth stage (three stage of cleaners on the rougher concentrate), it reached to about 70% (69.42%), that is desirably more than 60% which is the commercial grade.



	1 stage	2 stage	3 stage	4 stage
🟦 Grade	12.4	39.39	57.33	69.42
🟪 Recovery	94.07	72.97	75	40.03

Figure 9. Effect of cleaning stages on lead grade and recovery.

3.1.12. Study of process kinetic

To study the floating rate of mineral particles at a time unit through flotation process, froth was collected in time intervals. The kinetic diagram was then drawn as cumulative recoveries versus time intervals. The cumulative recovery had an upward trend. In the first 6 minutes, the rate of increase was low. After 6 minutes a sudden increase occured in recovery, and it slowed down after 12 minutes, and from 18th minutes onwards the recovery was approximately constant (Figure 10). As a result, 18 minutes was a suitable time for froth taking.

The remarkable increase in recovery after 6 minutes could be the result of sulfur consumption by the dissolved lead in the cell; usually a part of lead preticipate as lead sulfide in pulp. By adding the sulfidation agent, the sulfide ions were consumed by the dissolved lead particles. These colloidal particles preticipate on cerrusite surface and interact with collector rapidly. As a result, at the beginning of froth taking process, a remarkable increase was observed at the flotation curve. There existed an assumption which was the conditioning time of sodium silicate causes a delay in the flotation of lead particles. During the experiments with no dispersant, this delay did not occur.

By passing the curve from the obtained data of kinetic experiment, the kinetic factor of the examined material could be calculated. The equation introducing the curve is: $y = 94.06\left(1 - e^{-0.206t}\right)$. As a result, the rate constant of equation for the Changarzeh lead sample was estimated k=0.206 min.$^{-1}$, and recovery at the practical infinity was equal to 94.06% (Figure 11).

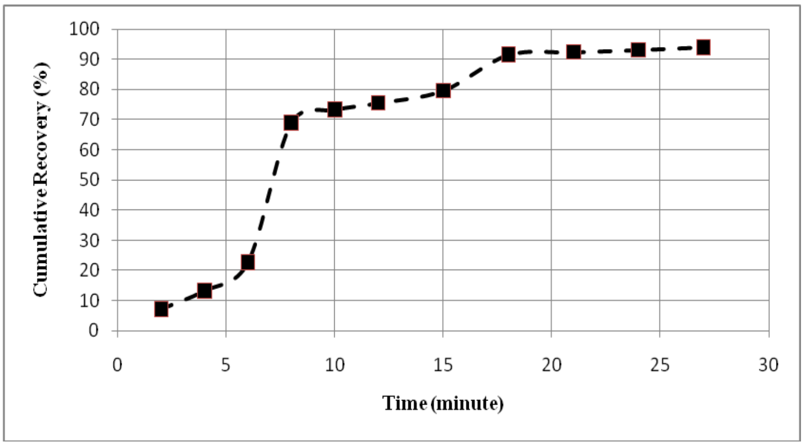


Figure 10. The flotation time versus cumulative lead recovery in kinetic experiment.

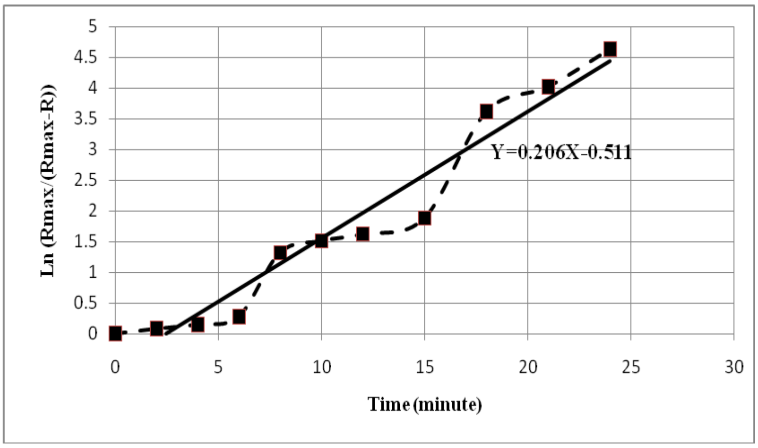


Figure 11. The kinetic curve used in rate constant estimation.

3.2. Gravity methods

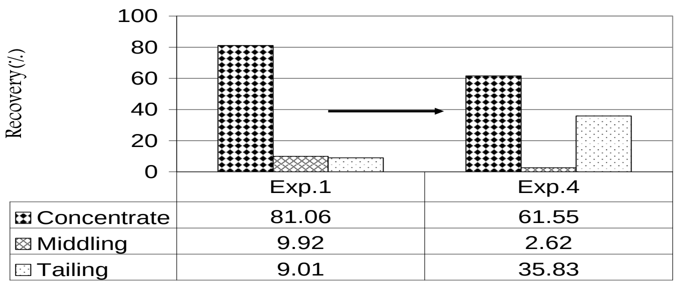
The preliminarily experiments were conducted to determine if the gravity methods are suitable for pre-concentrate production. Then the grade and recovery variation versus effective parameters were investigated.

3.2.1. Shaking table experiments

The results of microscopic studies demonstrated that the valuable particles liberated at 212 microns. Also it is known that the more limited the table feed is, the more will be the table efficiency. So the sample was divided in various fractions before testing with table.

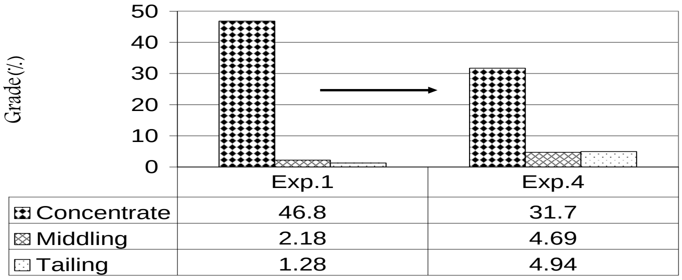
For the fraction of -850+150 microns, the following comparative experiments were established. In the first experiment, the slope of 12% and the feed water of 10 lit/min. lead to the highest grade and recovery of 46.8% and 81%, respectively. To increase the lead grade, in the second experiment the table slope was decreased from 12% to 10% and the water of feed and table water were increased to 10 and 12 lit/min. respectively. These changes lead to a decrease in recovery and grade (Figures 12 and 13). As a result, the first experiment condition proved to be the optimum. The obtained grade can reach 50% by a cleaning stage.

For the fraction of -1700+850 microns, considering the increase in particle size, the 3rd experiment was conducted by a slope of 13%, water of feed of 8 lit/min. and table water of 10 lit/min. As a result, a great amount of tail entered the concentrate and a low lead grade of 8% by the recovery of 63% was obtained. The microscopic studies demonstrated that the galena particles were not liberated in the middling and tailing sections. It indicated that the particles were not liberated at this size. In the 4th experiment, to attain a higher grade, the table slope was lowered to 12% and during the experiment the table water was increased to 12.5 lit/min.; an increase in concentrate grade was attained and a concentrate of 25% with a recovery of 54% of lead was achieved. Meanwhile, the grade of 25% of lead was not remarkable. As a result, this fraction was not suitable for table. Variations in grade and recovery can be seen in Figures 14 and 15.



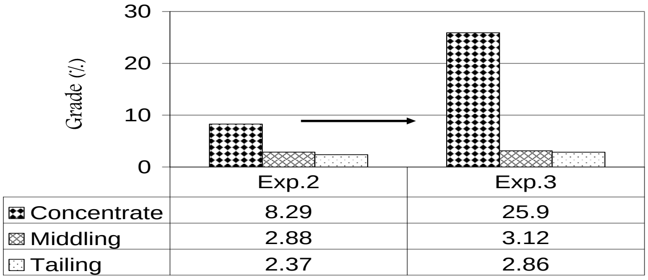
	Exp.1	Exp.4
▦ Concentrate	81.06	61.55
▨ Middling	9.92	2.62
⬚ Tailing	9.01	35.83

Figure 12. Table slope decrease, and water of feed and table water increase versus lead recovery.



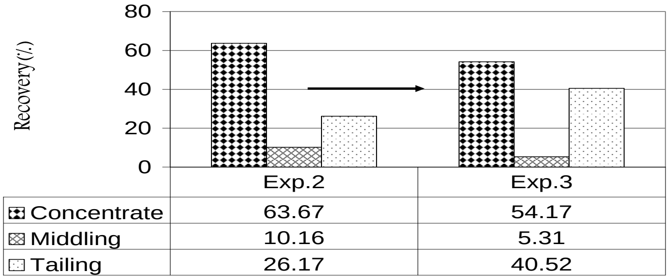
	Exp.1	Exp.4
▦ Concentrate	46.8	31.7
▧ Middling	2.18	4.69
☐ Tailing	1.28	4.94

Figure 13. Table slope decrease, and water of feed and table water increase versus lead grade.



	Exp.2	Exp.3
▦ Concentrate	8.29	25.9
▧ Middling	2.88	3.12
☐ Tailing	2.37	2.86

Figure 14. Table slope decrease and table water increase on lead grade (coarse particles).



	Exp.2	Exp.3
▦ Concentrate	63.67	54.17
▧ Middling	10.16	5.31
☐ Tailing	26.17	40.52

Figure 15. Table slope decrease and table water increase on lead recovery.

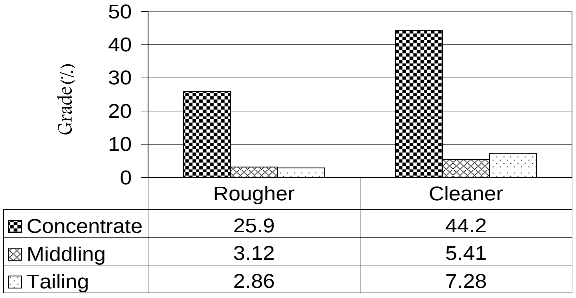
3.2.2. Study of cleaning stages

To study the effect of cleaning on rougher concentrate obtained from shaking table, the second experiment (fine fraction) and the third experiment concentrate (coarse fraction) were tested. The fine concentrate of the second experiment underwent a cleaning stage by a table slope of 12%, water of feed of 9 lit/min. and table water of 10 lit/min. for attaining a higher grade.

Cleaning the coarse concentrate by a table slope of 12%, water of feed of 10 lit/min. and table water of 14 lit/min. led to a higher grade. In both cases concentrate grade arrived at 50% of lead. Figures 16 and 17 present the comparative results.

3.2.3. Jig experiments

In jig size limits were -2360+850 and -850+300 microns, water flow was between 4-5 lit/min. and frequency of jig was between 80-270rpm. The effect of these parameters and the number of jig balls were studied on the separation of the particles. The best concentrate grade was 37.33% which came in the fraction of -2360, water flow of 4 lit/min. and the frequency of 269rpm accompanied balls. This grade was not an acceptable grade for jig. Also according to the low grade and the fine lead particles existing in feed and its loss in jig tail, jig was not recommended for pre-concentration production.



	Rougher	Cleaner
▦ Concentrate	25.9	44.2
▧ Middling	3.12	5.41
☐ Tailing	2.86	7.28

Figure16. Effect of cleaning on table product lead grade (coarse particles).

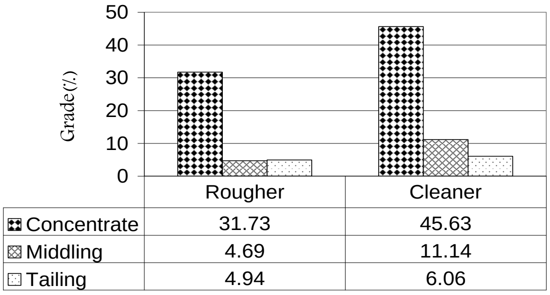


Figure17. Effect of cleaning stage on product lead grade (fine particles).

4. Conclusions

The Changarzeh low grade sulfide-oxide lead ore was studied, and mineral processing studies concentrated on increasing the lead grade. This sample had a lead grade of 2.3% and zinc grade of 1.92%, including dolomite, quartz, ankrite, gismondit, galena, cerrusite, tetrahedrite, anglesite, pyrite, calcopyrite, sphalerite, calcosite, calcite, albite and gypsum.

A series of experiments were carried out to determine the optimum dosage of effective parameters and the following conclusions were drawn.

- Maximum flotation recovery of the Changarze sulfide oxide ore was attained by cumulative flotation of the sulfide and oxide phases.

- 2000g/t of sodium sulfide was the most suitable amount for sulfidation of oxide minerals and the preparation time of 6 minutes was sufficient for sulfidation.

- Potassium amyl xanthate (Z11) as collector, showed better results comparing sodium isopropyl xanthate. The use of 200g/t was the suitable consumption for this collector.

- Among frothers, pine oil, MIBC, Aerofloat, MIBC had the most compatibility with the optimum test condition.

- Adding chemicals in stages for preventing sudden increase in pH and galena depression did not have a remarkable effect on increasing recovery.

- 500g/t of sodium silicate was useful for fine particles dispersion.

- By 3 cleaning stages, the concentrate grade increases up to 69.42%, that introduced a commercial product. The recovery was 40% which is acceptable for low grade ores.

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