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Technical Note / Teknik Not

An investigation on characterization and floation behavior of a transitional ore containing Galena and Pb Oxide minerals

Galen ve Pb oksit mineralleri içeren geçis zonu cevherinin karakterizasyonu ve flotasyon davranışının incelenmesi

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ABSTRACT

Flotation behavior of a lead ore sample containing both sulfide and oxide Pb minerals taken from transition zone was investigated. Detailed mineralogical analyses were conducted to determine the type of lead and gangue minerals. The results revealed that galena, cerussite and pulmbogummite were the major lead minerals and observed in close association. In the flotation tests, following determination of the optimum particle size, effects of sulfidization, type of collector, and frother were examined. Potassium amyl xanthate, a modified mercaptan (Aero 412), and and the frother (Dowfroth-250) produced the best results. Use of Flash flotation was tested to reduce the flotation time and recover the fast-floating liberated galena particles at coarse particle size. A locked cycle flotation test was conducted using the optimum flotation conditions and 50% lead containing concentrate was thought to be produced with 88.9% recovery under plant conditions.

Keywords: Cerussite, Galena, QEMSCAN, Sulfidization, Flash flotation, Locked cycle flotation

ÖZ

Bu çalışmada hem sülfürlü hem de oksit yapıdaki Pb minerallerini barındıran, geçiş zonuna ait bir kurşun cevherinin flotasyon davranışı incelenmiştir. Detaylı bir mineraloji analizi yapılarak kurşun ve gang mineralleri tespit edilmiştir. Kurşun minerallerinden galen ve serüzit ile birlikte plumbogummit belirlenmiş olup, oksit ve sülfürlü kurşun minerallerinin birlikte bulunduğu bir yapı görülmektedir. Flotasyon testlerinde, öncelikle tane boyutunun etkisi incelenmiş, daha sonra sülfürleştirici, toplayıcı ve köpürtücü reaktif türlerinin kurşun flotasyonu üzerindeki etkisi araştırılmıştır. Flotasyon testlerinde potasyum amil ksantat, modifiye merkaptan (Aero 412) ve köpürtücü (Dowfroth-250) birlikte kullanıldığı durumda en yüksek verime ulaşılmıştır. Flotasyon süresini kısaltmak ve görece iri boyutta serbestleşen kurşun tanelerinin yüzdürülmesi amacıyla flaş flotasyonun etkisi araştırılarak en uygun açık devre tasarımı yapılmıştır. En iyi flotasyon performansının elde edildiği koşullarda kapalı devre flotasyon testi yapılmış ve tesis koşullarında %50 kurşun içerikli bir konsantrenin %88,9 verim ile üretilebileceği öngörülmüştür.

Anahtar Sözcükler: Serüzit, Galen, QEMSCAN, Sülfürleme, Flaş flotasyon, Kapalı devre flotasyon

Introduction

Flotation is a widely used method in beneficiation of both oxide and sulfide type lead ores. The most widely used lead minerals in primary lead metal production after galena (PbS) are its secondary minerals that are anglesite ($PbSO_4$) and cerussite ($PbCO_3$).

There are specific distinctions in flotation of oxide and sulfide type lead ores. When compared with sulfide minerals, oxide minerals have higher solubility, which led to higher wettability character of surfaces. This situation makes flotation of oxide lead minerals challenging (Herrera-Urbina et al., 1998). In addition, slime coating is another effect with lower flotation recoveries due to their easy grindability character. Cerussite mineral – known as lead carbonate – has a soft structure and intends to get finer during grinding (Elizodo-Álvarez et al., 2020). Further, reagent consumptions increase, because of reactions between reagents

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and metal cations, which are easily dissolved from oxide minerals (Marabini et al., 2007; Abkhoshk et al., 2014). Another issue is the variability of flotation behaviors of ores having similar electrophoretic character from various locations (Fuerstenau et al., 1987).

On the other side, oxidation degree of galena surfaces may vary depending on the geological formation conditions. In case of partial oxidation of galena surfaces, certain promoters are necessary to be used. Depending on the oxidation degree, addition of sodium sulfide or sodium hydrosulfide before flotation is effective (Önal et al., 2005).

If most of the lead minerals are in oxide form, galena is floated at first, then, oxide flotation is performed. Oxide flotation can be carried out using fatty acids or amines, while sulfide flotation following sulfidization is preferred (Somasundaran ve Lou, 1999).

In the literature, collectors of isopropyl or amyl xanthate are suggested with AERO 404, 407 or 412 containing mercaptobenzothiazole and dithiophosphate mixture (Thomas, 2010). Generally, sulfidizing reagents are added as multistage at the rougher flotation stage. The dosage of the sulfidizing reagents varies between 500 and 2500 g/t (Önal et al., 2005).

Sodium sulfide or sodium hydrosulfide are used as sulfidizing reagents. Sulfidizing occurs, when the HS^-/S^{2-} ions cover the oxide surface and form metal sulfide shell (Fa et al., 2005). The reaction between cerussite and sodium sulfide are illustrated in Eq 1-4 (Wills and Napier-Munn, 2006).

$$Na_2S + H_2O \longrightarrow NaHS + NaOH \quad (1)$$

$$PbCO_3 + 3NaOH \longrightarrow H_2O + Na_2CO_3 + NaHPbO_2 \quad (2)$$

$$NaHS + NaHPbO_2 \longrightarrow 2NaOH + PbS \quad (3)$$

$$Na_2S + PbCO_3 \longrightarrow Na_2CO_3 + PbS \quad (4)$$

Sulfidizing reagents also prevents an increase in reagent consumptions by precipitating dissolved Pb^{2+} ions (Fuerstenau et al., 1987). However, using excessive amounts of sulfidizers have negative effect on flotation of oxide lead minerals (Bulatovic, 2010). It is known that sulfidization occurs following a reaction of between heterogenous solid-liquid phases resulted in a solid multi-layer cover on cerussite mineral (Liu et al, 2020). Therefore, dosage of sulfidizing reagent addition is critical. Overdose usage inhibits adsorption of collector on mineral surface. Further, high alkalinity leads to formation of high HS^-/S^{2-} concentration and causes lower recovery (Wills and Napier-Munn, 2006). To overcome these issues, studies have focused on investigating increasing the effect of sulfidizers using less dosages and preventing depressant effect. In addition, to collect more Pb ions in the electrical double layer, addition of NaCl salt was examined (Feng et al., 2015; Feng et al., 2016; Liu et al., 2019).

When the lead formations in the world is investigated, it can be clearly seen that oxide, transition and sulfide lead structures are observed in an order from top elevations to deeper levels. While flotation of oxide and sulfide lead ores can be performed in separate stages under different reagent conditions, flotation of transitional ores having a mix structure differs. Regarding their complex mineralogy, beneficiation step is a challenge. In industrial applications, there is a sequential method. Following galena flotation at the first stage, oxide lead is floated using sulfidizing agent. Both stages may require long flotation times, in addition, the particle liberation sizes for oxide and sulfide lead minerals can be different. These issues affect the plant capacity directly, where alternative process designs are necessary.

In the scope of this study, a transitional lead ore containing both oxide and sulfide minerals were subjected to mineralogy examination and obtaining lead concentrate with acceptable recovery and grade (~80% recovery and ~60% Pb grade) was investigated. Further, a process flowsheet is generated by applying a combination of flash and conventional flotation.

1. Material and method

The study was conducted on the samples taken from Karaganda, Kazakhstan. Following characterization of the ore, parameters for flotation process design were examined such as the effect of particle size, type of collector, frother, sulfidizing agent, promoter, flotation time, and flash flotation. These parameters were optimized regarding the results of rougher kinetic flotation tests. Cleaning stages revealed that the flotation flowsheet and final products were obtained after locked cycle flotation test.

1.1. Material

QEMSCAN (quantitative evaluation of minerals by scanning electron microscopy), SEM-EDS (Scanning Electron Microscopy/ Energy Dispersive X-Ray Spectroscopy) and XRD (X-Ray Diffraction) analyses were made in the laboratories of ALS, Australia. Following classification of the ore sample by grinding below 100 µm as; +100, -100+75, -75+36, -36+20, and -20 µm, mineralogy analyses were held. Chemical analyses were conducted using the ICP (inductively coupled plasma) and LECO CS844 (C-S analysis) devices in ESAN laboratories.

1.2. Method

Primary grinding was performed using standard Bond ball mill, while regrinding was conducted in planetary type ball mills with 20 cm. Flotation tests were carried out utilizing Denver D-12 type flotation machine. In the rougher stages, experimental constants were 2 kg of sample, 30% solids ratio (weight by weight), and 4.5 L cell volume. For the cleaning stages, 1.5 L cells were used, while the flash flotation was performed in 2.5 L cell with a particle size fraction of -400 µm.

Initial sulfide and oxide rougher flotation tests were carried out for 10 min (2+4+4), respectively. Equal amounts of reagent addition was provided. Regarding the conditioning times, 10 min for sulfidizers, 2 min for collectors and promoters, and 1 min for frothers were kept constant.

The locked cycle test was completed till obtaining a consistent final concentrate grade in the last three concentrates by recycling the water. While the natural pH was nearly 7, the value increased slightly after reagent addition. pH adjuster was not used.

In determining the primary grinding size, -130, -90, and -63 µm fractions were tested. For the secondary grinding, d_{80}: 40 µm was investigated. The reagents of MIBC, Na_2S, NaHS, KAX, and SIBX were supplied by ECS Kimya. Oreprep F-549, Aero 412, and Aero 6493 were SOLVAY products and Dowfroth 250 (DF-250) is Dow product.

2. Findings and discussion

2.1. Mineralogical characterization

Fractional chemical analyses of the sample are illustrated in Table 1. It is understood that Pb grade increases as the particle size decreases. Specifically, Ag is mostly accumulated in the finest fraction.

In addition, mineral distribution by weight is given in Table 2. Regarding the results, the major Pb component was determined as cerussite, while galena was found as half of cerussite by weight. Further, a rare Pb mineral type of plumbogummite ($PbAl_3(PO_4)(PO_3OH)(OH)_6$) was detected, which was found as much us galena amount. Also, another type of Pb phase of Pb-silicate was observed by the QEMSCAN. Flotation of these both phases are nearly impossible.

When the Pb grades are distributed regarding their stoichiometry, it was revealed that 30.5% and 55.5% of the Pb was associated with galena and cerussite, respectively. Table 3 shows the liberation of lead minerals. According to the distribution of galena and cerussite values, liberation cannot be fully provided even for 75 µm.

Table 1. *Chemical analyses of the raw ore sample*

Size fraction	Amount	Pb		Ag		S		C	
µm	%	G, %	D, %	G, ppm	D %	G, %	D, %	G, %	D, %
+100	17.8	1.18	5.8	7.53	6.0	0.25	8.4	0.06	7.4
-100 +75	8.9	2.05	5.1	9.25	3.7	0.36	6.1	0.07	4.4
-75 +36	16.2	2.84	12.8	12.83	9.3	0.54	16.6	0.11	12.6
-36 +20	41.2	3.90	44.8	16.57	39.8	0.60	46.8	0.17	49.0
-20	15.9	7.13	31.5	41.98	41.2	0.74	22.2	0.24	26.6
Total	100.0	3.59	100.0	17.74	100.0	0.53	100.0	0.14	100.0

* G: Grade – D: Distribution

Table 2. *Distribution of minerals by weight*

Mineral Group	Distribution by weight, %					
	+100 µm	-100/+75 µm	-75/+36 µm	-36/+20 µm	-20 µm	Combined
Galena	0.43	0.66	1.30	1.39	2.12	1.26
Cerussite	0.79	1.62	1.92	2.90	4.92	2.57
Pb-phospate (pyromorphite-type)	0.07	0.08	0.11	0.11	0.19	0.11
Pb-Al-phospate (plumbogummite-type)	0.23	0.38	0.39	0.90	2.90	0.97
Pb-silicate intergrowths	0.37	0.49	0.47	0.90	3.07	1.04
Other Pb intergrowth	0.11	0.23	0.21	0.54	0.86	0.43
Sphalerite and other Zn minerals	0.03	0.03	0.05	0.05	0.10	0.05
Chalcopyrite and other Cu-sulphides	0.01	0.03	0.01	0.02	0.04	0.02
Pyrite	0.32	0.52	0.55	0.61	0.53	0.53
Other sulphides/intergrowths	0.01	0.03	0.01	0.02	0.01	0.01
Quartz	21.6	21.5	22.6	22.6	17.1	21.5
Albite	0.05	0.09	0.14	0.30	0.29	0.21
K-feldspar	68.5	68.0	65.4	61.6	54.0	62.8
Muscovite/biotite	5.11	4.51	5.15	4.90	7.83	5.41
Other silicates/intergrowths	0.17	0.24	0.18	0.61	1.81	0.62
Rutile/anatase	0.20	0.36	0.31	0.44	0.89	0.44
Calcite and other carbonates	0.17	0.19	0.25	0.43	0.34	0.32
Siderite/goethite	0.62	0.50	0.46	0.69	0.87	0.65
Barite	0.18	0.31	0.33	0.43	0.89	0.43
Steel/Fe-alloy	0.99	0.09	0.09	0.27	0.59	0.40
Other minerals	0.06	0.19	0.08	0.30	0.66	0.27
Total	100.0	100.0	100.0	100.0	100.0	100.0

Table 3. *Combined distributions of galena and sphalerite liberation*

Liberation class	Combined distributions of cerussite and galena	Distribution by weight, %					Combined
		+100 µm	-100/+75 µm	-75/+36 µm	-36/+20 µm	-20 µm	
Well liberated	–100 - 90 %	20.0	37.2	53.8	39.4	31.5	37,9
High grade middlings	–90 - 60 %	16.4	25.6	22.4	41.5	46.6	38,1
Medium grade middlings	–60 - 30 %	12.8	13.7	9.9	12.1	16.4	13,2
Low grade middlings	–30 - 10 %	26.0	13.5	8.6	4.3	4.4	6,6
Locked	< 10 %	24.8	10.0	5.3	2.7	1.1	4,2
Total		100,0	100.0	100.0	100.0	100.0	100.0

2.2. Effect of particle size on Pb flotation

It is known that usage of promoters in the oxide flotation stages is necessary for high recoveries. To determine the optimum primary grinding size, 5 g/t A412 and 20 g/t KAX were added at each stage and 1000 g/t NaHS was used following the third stage.

As shown in Figure 1, the general recoveries were found to be nearly the same, although sulfide stage recoveries were lower for the finer size fractions of -90 and -63 µm.

NaHS was added after 10 min of sulfide flotation. Since the galena and cerussite particles were associated together at coarse fractions, the flotation test performed using 130 µm resulted in collecting of more cerussite in galena stage. more.

Figure 2 demonstrates the flotation time and recovery curve. It is understood that decreasing the particle size enhanced the liberation of galena and cerussite associated particles. While the recovery of 130 µm was higher in sulfide flotation, final recoveries were close to each other after sulfidizing reagent addition.

Besides, the grade vs. recovery curve shows an increase in Pb grades, as the particle size was decreased. Still, final grades of the concentrates were similar with high metal recoveries. Therefore, primary grind size of 130 µm was determined as the optimum for the transition ore sample. However, regrinding was found to be necessary to increase the Pb grade. Hence, just 10% of the feed material by weight would be reground, rather than the whole mass.

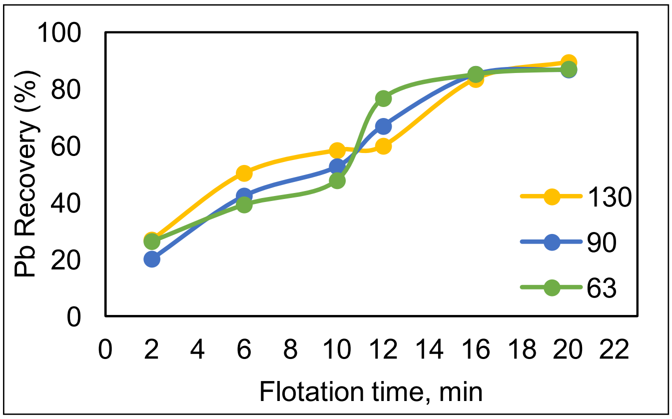

Figure 1. *Flotation time and Pb recovery relationship regarding grinding size*

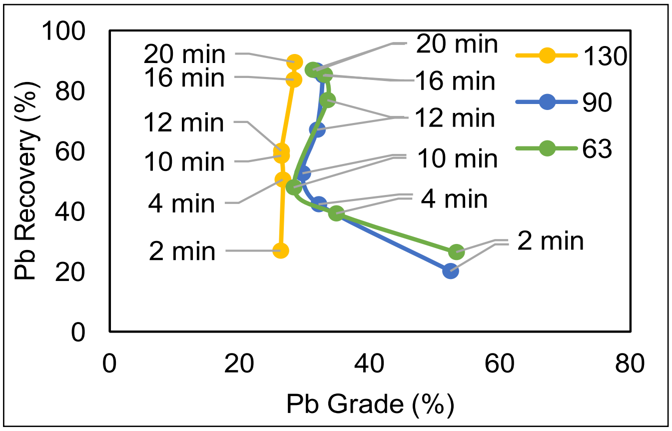

Figure 2. *Pb recovery and grade relationship regarding grinding size*

2.3. Effect of frother type

In this study, it was observed that the reagents used in the rougher stages mentioned in the previous section caused a decrease in surface tension. The best method to determine the effect of frothers is industrial applications. Nevertheless, the effect of frothers were decided to be investigated at laboratory scale. MIBC (Methyl Isobutyl Carbinol), polyglycol type F-549 and DF-250 were tested. As seen in Table 4, the highest selectivity was achieved using MIBC, however the highest recovery was obtained in the presence of DF-250 with low selectivity. The reason for this result might be attributed to higher mass pull. Considering the bubble stability, DF-250 was selected as the optimum frother with the highest recovery and acceptable Pb grade in the rougher concentrate.

Table 4. *Effect of frother type on Pb grade and recovery*

Frother Type	Amount, %	Pb Grade, %	Pb Recovery, %
DF-250	10,0	30,3	76,3
F-549	8,5	34,7	73,3
MIBC	7,5	37,9	69,2

2.4. Effect of promoter and collector type

As collectors, KAX was tested comparing with SIBX, which has more convenient storage conditions. In addition, collector:promoter ratio was kept constant as 4:1. Aero 6493 (hydroxamate) and Aero 412 (Mercaptobenzothiazole and dithiophosphinate mixture) were used as promoters. Table 5 shows that KAX + Aero 412 combination provided the highest recovery. Although Aero 6493 increased the Pb recovery slightly, Pb grade decreased dramatically. In case of SIBX usage, it was understood that there was a decrease in mass pull and Pb grade of the concentrate. Moreover, the recovery is lower than the results of KAX usage scenario. Hence, KAX + Aero 412 was determined as the optimum.

Table 5. *Effect of collector and promoter type on flotation*

Collector+ Promoter	Amount, %	Pb grade, %	Pb Rec., %
KAX	9.4	35.1	82.9
KAX + Aero 412	11.3	28.3	89.1
SIBX + Aero 412	9.8	33.4	86.8
SIBX + Aero 6493	14.2	23.6	88.2

2.5. Effect of sulfidizers

Regarding the equations given in Equation 1-4, it was indicated that NaHS or Na_2S can be utilized as sulfidizers effectively. In this part of the study, the effect various additions of them on Pb flotation was examined. The results show that NaHS was more effective at lower dosages (Table 6). The findings given in literature supports this outcome, where high sulfidizer dosages negatively affect metal recoveries (Fuerstenau et al., 1987; Herrera-Urbina et al., 1999; Liu et al., 2020). Further, pH measurements were carried out during the tests. In the presence of Na_2S, pH increased up to 1 level, however, pH was nearly 9.5, when NaHS was used. Critically, it was observed that bubble stability decreased due to the increase in pH (Farrokhpay and Zanin, 2012).

Table 6. *Effect of sulfidizer on flotation*

Amount, g/t	Pb Recovery Increase, %		pH	
	Na_2S	NaHS	Na_2S	NaHS
600	11.1	14.5	9,7	9,5
800	12.9	26.9	10,0	9,5
1000	24.8	19.6	10,2	9,6
1200	17.5	-	10,6	

2.6. Effect of flash flotation

Up to this part of the study, it was determined that the flotation time for rougher stage was relatively long (20 min). Therefore, flash flotation was applied at a particle size fraction of d_{100}: -400 µm, in order to recover coarse galena particles. At the same time, it was an opportunity to prevent overgrinding of lead particles, which might lead to low recoveries. Thus, the effect of fine particles having slow flotation kinetic could partially be eliminated. Also, it was aimed to make a design including addition of the sulfidizer at the beginning the rougher flotation following flash flotation, where most of the galena was collected.

Acceptable recoveries were achieved even at coarse particle size (Figure 3). The first 60 sec was found to be the optimum flotation time, since mas pull decreased at the second 60 sec.

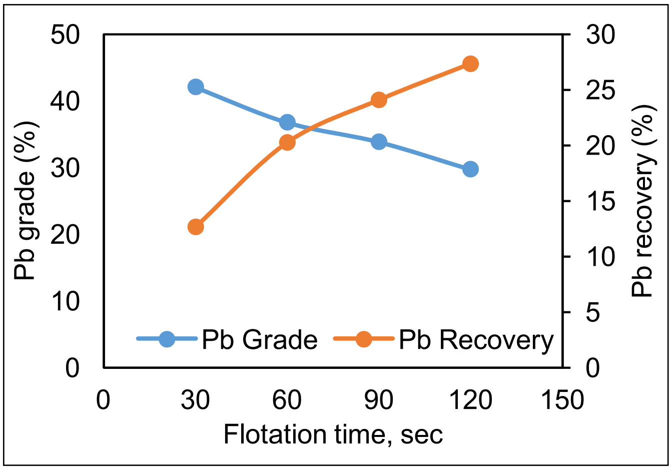

Figure 3. *Effect of flash flotation on Pb grade and recovery*

2.7. Effect of cleaning stages on the concentrate grade

In order to obtain saleable concentrate, the flash flotation and rougher flotation concentrates were combined and reground below 40 µm. This product was subjected to 3 stage cleaning flotation. The results are illustrated in Table 7.

Table 7. *Results of open cleaner test*

Product	Amount %	Pb grade %	Pb Rec. %
Cleaner 3 concentrate	3.5	63.13	57.9
Cleaner 3 tailing	1.6	25.01	10.5
Cleaner 2 tailing	2.4	14.61	9.19
Cleaner 1 scv. con.	0.9	16.07	3.77
Cleaner 1 scv. tailing	6.3	6.35	10.4
Rougher tailing	85.3	0.37	8.28
Total	100.0	3.82	100.0

Regarding the results, 14% Pb was lost in the cleaner 1 tailing. Considering a locked cycle operation, this product might be a problem with high amount Pb, since it would be recycled to the rougher stage. Therefore, a scavenger stage was decided to be applied on this product.

The cleaner 3 concentrate contained 63% Pb, while the recovery was obtained as 58%. This result revealed that a locked cycle flotation test was planned without using depressants and recycling the products at their own stage.

2.8. Locked cycle flotation test

The aim of this test was to produce a final concentrate and determine the grade and recovery. Following the flowsheet demonstrated in Figure 4, the results given in Table 8 were achieved.

As is seen, a Pb concentrate containing 50.36% Pb with a recovery of 89% was obtained.

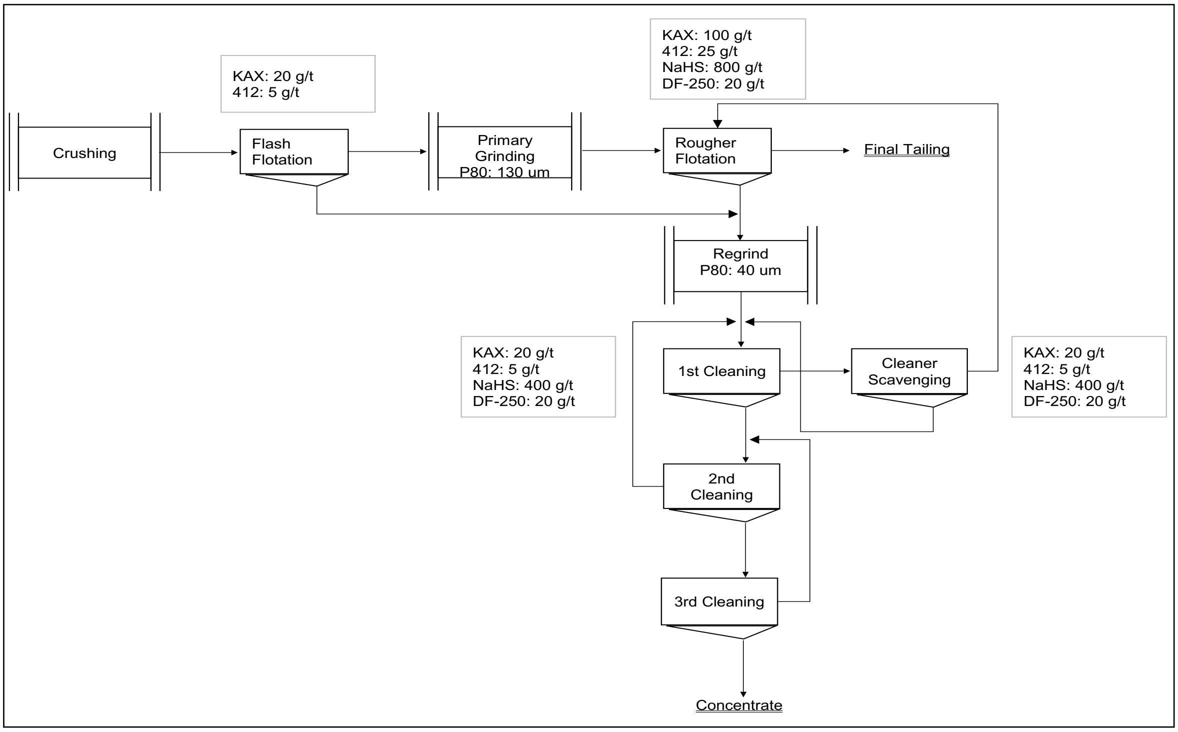

Figure 4. *Flowsheet of the locked cycle flotation test*

Table 8. *Results of locked cycle flotation test*

Product	Amount, %	Pb grade, %	Pb Recovery, %
Concentrate	6.34	50.36	88.9
Tailing	93.7	0.43	11.1
Total	100.0	3.59	100.0

2.9. Discussion

When the sulfide minerals oxidized, secondary minerals are formed with different flotation behavior. These mineral forms may vary regarding the mine deposits (Fuerstenau et al., 1987). Therefore, specific flowsheets are required for each of ores. However, applying different flowsheets for the different type of ores from the same deposit may increase the complexity of the operation. In this way, it is a difficulty to find the required masses for plant feeds.

In this study, which was originated from such a case, the Pb ore containing high amount of secondary Pb mineral was beneficiated. By this means, evaluation of the oxide and sulfide mixture of transitional ore could be enabled. In the meantime, the outcomes of this experimental study made a significant contribution to the sustainability and life of mine.

On the other side, a highlight of the study – flash flotation – was expected to provide advantage in three ways: i) slime generating for the Pb particles was eliminated by multistage grinding, ii) flotation of coarse liberated galena particles were achieved with a quite short time. Thus, rougher flotation was begun directly with sulfidizer addition, iii) necessity of large flotation cell volumes was eliminated due to shortened rougher flotation time.

In the literature, there are many studies conducted with sulfide and oxide Pb ores. However, this study is the first example of generating a flotation circuit including flash and conventional methods for a natural mixture of these ore types (transitional ore). This finding was thought as to bring the novelty and importance of the study.

Results and suggestions

Beneficiation of a lead ore containing oxide and sulfide Pb was investigated. It was determined that the 85% of Pb in the ore sample was composed of cerussite and galena. However, the rest of the Pb was found in the form of quite rare minerals, which are difficult to recover. Nevertheless, since the lead minerals were associated together, flash and conventional flotation tests applied at particle sizes of 400 and 130 µm, respectively, resulted in 90% Pb recovery with 50% Pb grade. This concentrate was achieved using160 g/t KAX, 40 g/t 412, 800 g/t NaHS, and 80 g/t DF-250.

Particularly for this ore;

- The recovery for the rougher stage was independent from particle size. Flotation performances were nearly the same for all sizes, which revealed that lead minerals were exposed well for collector adsorption. However, regrinding was necessary to produce saleable concentrate.

- Frother type was an essential variable. Depending on the usage of other reagents, the interaction between frother them affected selectivity directly.

- As a usual utilization of promoter in flotation of non-oxide base metals, it was found to be a necessity in this case, too.

- It was observed that NaHS was more effective than Na_2S at lower dosages and for bubble stability.

- Optimization was completed using adequate dosages of sulfidizers. Utilizing additives such as NaCl, which has been suggested in the literature, was not required.

- By means of flash flotation, overgrinding of the ore was prevented and rougher flotation time was shortened.

- Regarding the open cycle flotation test, the results revealed that acceptable recovery and grade could be achieved without any depressant.

- The locked cycle flotation test was performed by cleaning the rougher concentrate for 3 stages. Distinctly from the common sulfide Pb concentrates, a 50% Pb containing concentrate was obtained, where recovery was determined as 89%.

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