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Bulk Flotation of Low Grade Refractory Sulfide Lead-Zinc Ore

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In this research, bulk flotation from a low-grade refractor lead-zinc ore containing 3.5 % Zn and 1 % Pb and 1.9 % Fe was studied. Due to the problems involving sequential flotation of finely disseminated lead-zinc sulfide ore, the production of bulk lead-zinc concentrate was practiced. Flotation parameters such as activator (CuSO₄) dosage, collector type [potassium amyl xanthate, sodium isopropyl xanthate] and dosage, flotation time, particle size and pH were investigated. Experimental results showed that collector dosage was the most significant factor affecting Zn and Pb recoveries. In the bulk flotation stage where 100 g/t CuSO_4 and 150 g/t potassium amyl xanthate were used (at pH 8 and 10 min flotation time) maximum Zn and Pb recoveries were obtained as 94 and 83 %, respectively.

Key Words: Flotation, Sphalerite, Galena, Sulfide ore.

INTRODUCTION

The flotation process has been widely used, for over a century, to quickly and efficiently separate valuable minerals from gangue minerals, based on the differences in their natural or induced hydrophobicity. The process as a whole is very complex but can be described as the overlap of three main principles i.e., physical phenomena, chemical control and mechanical factors¹. Flotation is strongly affected by many factors regarding the floated minerals such as grade, degree of liberation, surface properties etc., and many operating variables². It is possible to obtain good performance from a flotation plant, but it has proved difficult to obtain and sustain good performance. The industrial flotation performance can be detrimentally affected by inevitable changes in the feed stream in terms of flow rate, grade, particle size distribution, etc.³⁻⁵. Batch flotation tests have been carried out extensively to investigate the effect of various operating parameters on flotation performance⁶. The effect of particle size on flotation performance has been widely studied to date and many important physicochemical factors related to particle size have been identified7-18.

Metal sulfide minerals, for which this process was originally developed, are generally weakly polar in nature and consequently most have a hydrophilic surface⁵. Hence, collector molecules such as xanthates and dithiophosphates are normally used to increase hydrophobicity. A sulfide mineral sphalerite (ZnS) responds poorly to thiol collectors due to the relative instability of zinc-xanthate and is commonly treated with copper sulfate solutions to activate it. In the differential flotation of polymetallic sulfide ores, copper sulfides and galena are floated away from sphalerite under mildly alkaline conditions and sphalerite is floated following activation with copper sulfate¹⁹. The activation of sphalerite by metal ions such as Cd(II), Pb(II), Ag(II) and especially Cu(II) has been studied extensively^{5,20-36}.

Selective flotation of complex sulfide ores poses several difficulties due to mineralogical characteristics, incomplete liberation and flotation chemistry differences between the ores. Complex mineralogy, connected with difficult flotation chemistry, has hindered the economic beneficiation of several large ore bodies throughout the world. Sequential lead-zinc flotation processes generally use differential flotation to recover galena in the first stages of flotation, followed by sphalerite flotation. This is made possible through the application of appropriate regulators and depressants which allow the selective adsorption of collectors. In the flotation of low-grade ores, the optimum distribution of flotation reagents and the optimization of the process parameters are of special importance. Under these optimum conditions, it might be possible to develop a process flowsheet for economical sphalerite production from such ores³⁷. An alternative flowsheet practiced in several operating plants is the bulk flotation flowsheet. This flowsheet is advantageous when the selectivity between lead and sphalerite has deteriorated after using the sequential flotation method.

The current paper reports on the flotation of low grade lead-zinc ore with pyrite a content of 4 % is defined as finely disseminated sulfide ore from Konya region in Turkey. In this study, the effect of collector (potassium amyl xanthate) and activator (CuSO₄) dosage, pulp pH, flotation time, particle size as well as various promoter reagents such as dithiophosphate (Aero 3477), phosphine-based collector (Aero 3418 A) and potassium ethyl xanthate (PEX) has been investigated on the recovery of bulk zinc-lead concentrate. Sequental lead-zinc flotation cannot be studied due to the fact that liberation of the individual minerals occurs below d_{80} approximately 16 mm for sphalerite and 10 µm for galena.

EXPERIMENTAL

Low grade sulfide Pb-Zn ore containing 3.5 % Zn, 1 % Pb and 1.9 % Fe from Konya region in Turkey was used in the experiments. X-ray diffraction analysis, scanning electron microscopy, energy dispersive X-ray spectroscopy analyses were performed for ore characterizations. It was determined by the X-ray diffraction analysis that the ore contains sphalerite, galena and pyrite as metallic minerals and mica group minerals, calcite, dolomite and quartz as gangue minerals. The chemical composition of the ore is given in Table-1.

TABLE-1 CHEMICAL COMPOSITION OF THE ORE							
Component	(%)	Element	(%)	Element	(%)		
Na ₂ O	0.01	Pb	1.01	Со	< 0.002		
MgO	10.04	Zn	3.48	Mn	0.04		
Al_2O_3	4.70	Fe	1.90	С	7.68		
SiO ₂	16.74	Cu	0.003	S	4.05		
K_2O	1.23	Cd	0.03	Ag (ppm)	3.70		
CaO	20.69	Ni	< 0.002				

Each ore sample with an amount of 500 g was dry-milled by using laboratory ball mill in the manner that 80 % (d_{80}) is below predetermined particle size. The slurry including 30 % solid by weight prepared by using deionized water was transferred directly to the flotation cell. Flotation experiments were carried out using a Denver D12 type laboratory flotation machine with 1 L capacity cell. In order to eliminate any differences in redox potential in each test, the pulp was agitated at a rate of 1500 rpm for a period of 10 min. After adjusting the pH of the pulp by using Na₂CO₃, predetermined amount of depressant, activator and collector were added respectively and each reagent were conditioned for 3 min. Then, frother (methyl isobutyl ketone, MIBK) was conditioned for 2 min before the air entered the pulp and flotation was carried out for 4 min. The froth was scrapped in each 5 s at a constant depth so that a bulk concentrate was recovered over the 4 min period. After flotation, the concentrates and tailings were filtered and dried for chemical analysis and mass calculation.

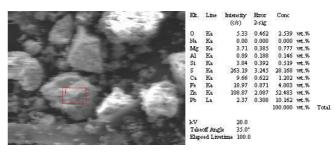
In this study, the flotation collectors (potassium amyl xanthate-PAX, potassium ethyl xanthate-PEX and sodium isopropyl xanthate-SIPX) and the frother (methyl isobutyl ketone-MIBK) and the depressant (sodium silicate) used in this study were of industrial grades. The other collectors, aerophine 3418A and aero 3477 were provided from Cytec Inc. All other reagents including copper sulphate and sodium carbonate were of analytical grade.

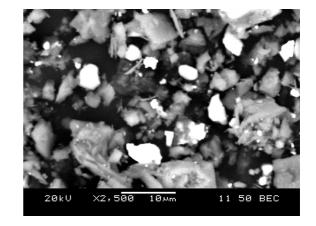
RESULTS AND DISCUSSION

Effect of particle size on bulk flotation: In order to determine the mineral liberation degrees, mineral liberation analysis (MLA), which is SEM based image analysis was used.

Table-2 indicates the liberation degrees of sphalerite and galena. Particles in the range of 80-100 % liberation class are commonly presumed as liberated. It is obvious from Table-2 that a significant amount of the sphalerite and galena are not liberated even at a fine particle size (-38 μ m). Furthermore, result of SEM based microscopic analysis indicates that the galena particles were disseminated in sphalerite particles (Fig. 1). The liberation analysis indicates that galena mineral is liberated at a relatively finer size compared to sphalerite. Consequently, bulk flotation was decided as the most proper method for recovery of zinc and lead.

TABLE-2							
MINERAL LIBERATION ANALYSIS RESULTS							
Size Fraction (mm) —	Liberation (%)						
	Sphalerite	Galena					
-2+1	12.81	3.28					
-1 + 0.5	11.91	2.26					
-0.5 + 0.3	30.32	29.38					
-0.3 + 0.1	23.06	19.72					
-0.1 + 0.075	44.06	43.33					
-0.075 + 0.053	42.34	34.94					
-0.053 + 0.038	52.14	41.05					
- 0.038	65.42	55.82					





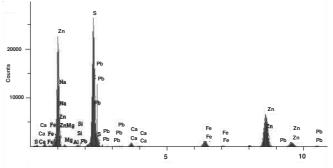


Fig. 1. SEM images and EDS analysis of ore

Size (µm) -	Collector		Additional	пЦ	$CuSO_4$	Solid	Flot. time	Recovery (%)	
	Туре	(g/t)	collector	pН	(g/t)	Ratio	(min.)	Zn	Pb
-300	Potassium amyl xanthate	100	-	8.0	400	%30	4	80.57	67.98
-150	Potassium amyl xanthate	100	-	8.0	400	%30	4	82.00	66.05
-106	Potassium amyl xanthate	100	-	8.0	400	%30	4	86.84	75.4
-75	Potassium amyl xanthate	100	-	8.0	400	%30	4	86.81	73.2
-53	Potassium amyl xanthate	100	-	8.0	400	%30	4	81.36	62.18
-106	Potassium amyl xanthate	100	-	8.0	400	%30	4	85.97	71.9
-106	Potassium amyl xanthate	50	-	8.0	400	%30	4	68.05	39.7
-106	Potassium amyl xanthate	150	-	8.0	400	%30	4	90.23	76.1
-106	Potassium amyl xanthate	200	-	8.0	400	%30	4	91.03	77.5
-106	Potassium amyl xanthate	150	PEX	8.0	400	%30	4	90.01	76.5
-106	Potassium amyl xanthate	150	3418A	8.0	400	%30	4	88.97	76.6
-106	Potassium amyl xanthate	150	3477	8.0	400	%30	4	90.16	78.9
-106	Potassium amyl xanthate	150	-	5.0	400	%30	4	90.72	71.0
-106	Potassium amyl xanthate	150	-	6.0	400	%30	4	89.02	70.2
-106	Potassium amyl xanthate	150	-	7.0	400	%30	4	90.72	72.7
-106	Potassium amyl xanthate	150	-	9.0	400	%30	4	91.82	79.4
-106	Potassium amyl xanthate	150	-	10.0	400	%30	4	92.11	79.3
-106	Potassium amyl xanthate	150	-	8.0	0	%30	4	85.94	78.2
-106	Potassium amyl xanthate	150	-	8.0	50	%30	4	90.46	79.5
-106	Potassium amyl xanthate	150	-	8.0	100	%30	4	92.05	79.7
-106	Potassium amyl xanthate	150	-	8.0	200	%30	4	91.37	77.9
-106	Potassium amyl xanthate	150	-	8.0	100	%20	4	89.89	75.2
-106	Potassium amyl xanthate	150	-	8.0	100	%40	4	91.60	78.5
-106	Potassium amyl xanthate	150	-	8.0	100	%30	2	74.25	65.8
-106	Potassium amyl xanthate	150	-	8.0	100	%30	6	92.45	81.5
-106	Potassium amyl xanthate	150	-	8.0	100	%30	8	93.22	82.6
-106	Potassium amyl xanthate	150	-	8.0	100	%30	10	93.63	83.3

In order to determine the optimum particle size, ore samples were grinded to below 300, 150, 106, 75 and 53 μ m. The best recovery results are obtained for Zn and Pb as 87 and 75 % respectively at -106 micron size. The difference in recovery percentages of sphalerite and galena minerals could be attributed to the differences in the degree of liberation from gangue minerals. Feed size is known to have a profound effect on flotation and there is a size range where most minerals float best. Recovery problems increase with finely disseminated ores where there is close mineralogical association between sphalerite, galena and gangue minerals. In order to achieve high recoveries and acceptable concentrate grades, grinding is a key factor. However, grinding the fine sizes introduces other problems for flotation process as high plant running costs since the energy used for grinding increases and more reagents due to the increase in particle surface areas are required for flotation (Table-3).

In Fig. 2, metal recoveries are plotted as a function of flotation feed size. As it is seen in the Fig. 2, zinc and lead recoveries are increasing while the flotation feed size is decreasing. However, Zn and Pb recoveries are decreasing when the smaller feed size (-53 μ m) is used. Fig. 2 indicates that -106 μ m is the best feed size for bulk flotation of lead-zinc ore.

In the following flotation tests, dry milled materials having particle size distribution of 90 % minus 100 μ m, 80 % minus 75 μ m, 50 % minus 18 μ m were used (Fig. 3). Particle size distribution analyses were accomplished by Horiba Partica LA-950V2 model laser scattering particle size distribution analyzer and particle size distribution graph of grinded ore sample is presented in Fig. 3.

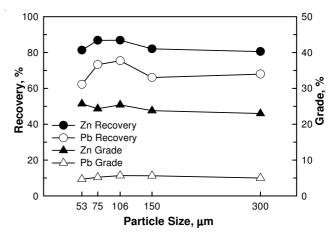
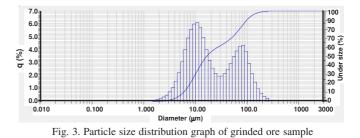


Fig. 2. Effect of particle size (pH: 8; Na₂SiO₃: 800 g/t; CuSO₄: 400 g/t; PAX: 100 g/t; MIBK: 50 g/t; Solid Ratio: 30 %; Flotation time: 4 min)



Effect of collector amount: In this stage, effect of potassium amyl xanthate amount on Pb-Zn grades and recoveries was investigated by using potassium amyl xanthate as the main collector. In order to determine optimum amount of collector, between 50 to 200 g/t of collector were experienced in these tests and obtained test results are given in Fig. 4. Consequently, Pb and Zn recoveries increased with increasing amount of collector up to 150 g/t. However, there was almost no change over 150 g/t collector consumption. In the test where 150 g/t of potassium amyl xanthate was used, Pb-Zn recoveries were 76 and 90 % and grades were 5 and 21 % respectively.

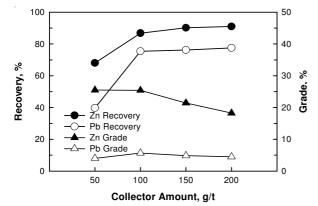


Fig. 4. Effect of collector amount (Particle size: -106 μm; pH: 8; Na₂SiO₃: 800 g/t; CuSO₄: 400 g/t; MIBK: 50 g/t; Solid Ratio: 30 %; Flotation time: 4 min)

Effect of additional collector type: In these tests, potassium ethyl xanthate (PEX), Aerophine 3418A (sodium-diisobutyl dithiophosphinate) and Aeropromoter 3477 (dithiophosphate) were experienced as collectors in addition to potassium amyl xanthate. The amount of potassium amyl xanthate was 150 g/t and the amount of additional collector was 75 g/t. Use of additional collectors had no important effect on zinc and lead recoveries (Fig. 5).

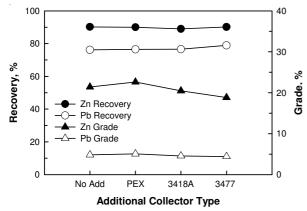


Fig. 5. Effect of additional collector type (Particle size: -106 μm; pH: 8; Na₂SiO₃: 800 g/t; CuSO₄: 400 g/t; PAX: 150 g/t; MIBK: 50 g/t; Solid Ratio: 30%; Flotation time: 4 min.)

Effect of pH: Six diffeent pH values were studied for the activation and flotation of sphalerite and galena. The results obtained from these experiments are given in Fig. 6. While there was no significant effect of pH for sphalerite recovery, the best results for galena were obtained in the pH range of 8-9. It was also observed that mica group minerals were floated and flocculated at higher pH values. In order to avoid from negative effects of these minerals during cleaning flotation, keeping the pH in the range of 8-9 will be appropriate.

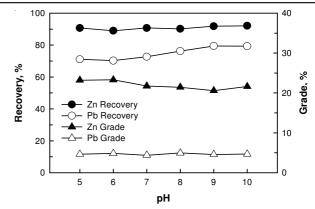


Fig. 6. Effect of pH (Particle size: -106 μm; Na₂SiO₃: 800 g/t; CuSO₄: 400 g/t; PAX: 150 g/t; MIBK: 50 g/t; Solid ratio: 30 %; Flotation time: 4 min)

Effect of sphalerite activator: Potassium amyl xanthate was used as the collector at a dosage of 150 g/t in amounts of 0, 50, 100, 200, 400 g/t copper sulfate was added as the activator and the frother (methyl isobutyl ketone) was kept constant at 50 g/t during these tests.

Fig. 7 shows the flotation of sphalerite as a function of copper sulfate concentration at pH 8. In the case of no CuSO₄ addition, the recovery of sphalerite was relatively high (about 85 %). The activation of sphalerite (no activator addition) may be explained by the fact that disperse fine galena particles are prone to surface oxidation and, thus they release Pb²⁺ ions in solution. Lead cations are well-known sphalerite activators³⁸. Sphalerite recovery increased up to 100 g/t of CuSO₄ usage while increased collector addition from 100 to 400 g/t did not significantly improve the maximum recovery. The addition of copper sulfate increased the sphalerite recovery, suggesting the activation of sphalerite by copper ions and formation of hydrophobic species on the sphalerite surface^{39,40}. The flotation performance of sphalerite depends on the level of surface activation. Fig. 7 indicates that the addition of high amount of CuSO₄ slightly decreased the galena recovery. At 100 g/t of copper sulfate, 92 % of sphalerite and 80 % of galena was recovered.

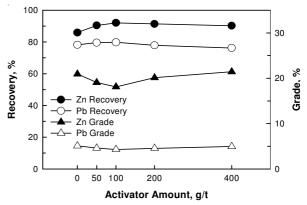


Fig. 7. Effect of CuSO₄ amount as activator (Particle size: -106 μm; pH: 8; Na₂SiO₃: 800 g/t; PAX: 150 g/t; MIBK: 50 g/t; Solid Ratio: 30 %; Flotation time: 4 min.)

Effect of solid ratio: Three solid ratios were studied for the flotation of sphalerite and galena. The results obtained are given in Fig. 8. Accordingly, sphalerite and galena recoveries

have increased up to 30 % solid ratio, the recoveries have remained unchanged in higher solid ratio. Also considering water consumption, solid ratio of 30 % will be appropriate in this flotation process.

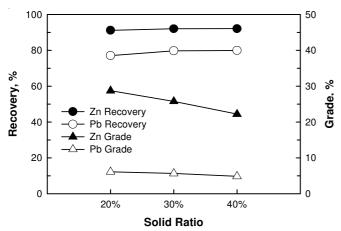


Fig. 8. Effect of solid ratio (Particle size: -106 μm; pH: 8; Na₂SiO₃: 800 g/ t; CuSO₄: 100 g/t; PAX: 150 g/t; MIBK: 50 g/t; Flot. Time: 4 min)

Galena, which is liberated in fine particles, was lost by desliming. When performed of feed desliming by 10 % in weight, losses of sphalerite and galena have been approximately 10 and 26 %, respectively. Therefore, feed desliming was not performed before flotation.

Effect of flotation time: In order to determine the optimum flotation time, kinetic flotation tests were performed by depending on flotation duration. The results obtained are presented in Fig. 9. As can be seen in Fig. 9, sphalerite and galena recoveries were increased in first four minutes, remained almost unchanged later. According to these results, the flotation time of 4 min is sufficient for rougher flotation stage.

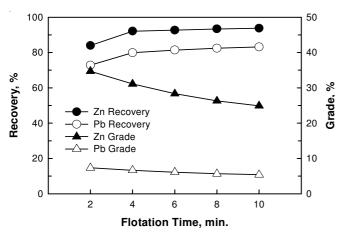


Fig. 9. Effect of flotation time (Particle size: -106 μm; pH: 8; Na₂SiO₃: 800 g/t; CuSO₄: 100 g/t; PAX: 150 g/t; MIBK: 50 g/t; Solid ratio: 30 %)

Conclusion

In this study, parameters affecting flotation process were investigated. Bulk lead-zinc flotation was applied to the ore due to the fact that liberation of the individual minerals occurs below d_{80} approximately 16 µm for sphalerite and 10 µm for galena. Overall flotation conditions and the test results for bulk flotation are given in Fig. 10 and Table-4. At optimum flotation conditions a rougher concentrate assaying 18 % Zn, 4.3 % Pb with recovery of 92 % Zn and 80 % Pb was obtained. To produce saleable sphalerite concentrate, a few stages of cleaning flotation were carried out the bulk concentrate. After 4 cleaning stages, clean bulk concentrate assaying 40 % Zn, 8.5 % Pb with recovery of 86.5 % Zn and 68 % Pb was produced. This concentrate is suitable for zinc extraction using direct atmospheric leaching process. According to the test results, a general flowsheet (Fig. 10) was developed including 1 rougher, 4 cleaning and 1 scavenging flotation stages.

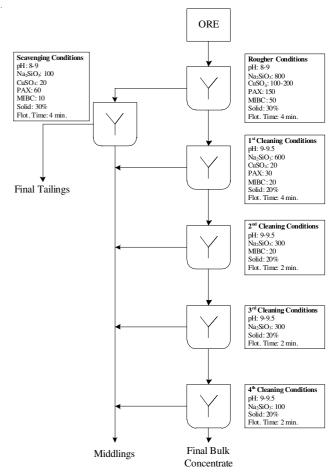


Fig. 10. General flowsheet of the flotation tests (all reagent dosages are in g/t)

BULK FLOTATION RESULTS IN OPTIMUM CONDITIONS								
Products	Weight	Grade	(%)	Recovery (%)				
Floducts	(%)	Zn	Pb	Zn	Pb			
Final bulk cons.	8.01	40.33	8.49	86.43	68.12			
Total middlings	5.84	3.52	1.98	5.50	11.58			
Final tailings	86.15	0.35	0.24	8.07	20.30			

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