

Journal of Mining & Environment, Vol. 9, No. 4, 2018, 1019-1033. DOI: 10.22044/jme.2018.6760.1498

# A comparative study of two-stage flotation of Zn and Pb oxide minerals using anionic, cationic, and mixed (cationic/anionic) collectors

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### Abstract

A lead-zinc carbonate ore sample containing 2.5% Pb and 9.39% Zn was used in this research work. The sample was prepared from the Darreh-Zanjir mine located in the Yazd province (Iran). Influences of the influential factors on flotation of smithsonite and cerussite were investigated. Among the different parameters involved, dosages of the dispersant, depressants, sulfidizing agent, and collectors de-sliming prior to lead or zinc flotation were essential for the effective recovery and grade of the Zn and Pb flotation concentrates. In addition, the anionic, cationic, and mixed (cationic/anionic) collectors were employed for flotation of smithsonite. The results of reverse and cumulative flotation of both Zn and Pb were relatively low in comparison with the direct process without depressant. Flotation of smithsonite using mixed collectors (Armac C+KAX) showed promising results. Also dosages of chemicals in the cleaning stage for the Zn and Pb grade and recovery of 49.82% and 60.06%, respectively, and smithsonite concentrate with Zn grade and recovery of 35.47% and 68.56%, respectively, were obtained under the optimal conditions. Furthermore, kinetics of Zn and Pb oxide mineral flotations in the rougher and cleaner stages were studied, which showed that some kinetics models, especially the classical first-order model, could predict the flotation behaviour of the Zn and Pb oxide minerals.

Keywords: Flotation, Anionic and Cationic Collectors, Zinc and Lead Oxide Minerals.

### 1. Introduction

The huge amount of ores present in mines is globally affected by the environmental conditions (i.e. climate, geochemical, etc.); therefore, they are oxidized and changed into waste nearby the mines [1]. The effective concentration by flotation of mixed lead and zinc ores remains a challenge to mineral engineers and scientists. Zinc is often found in association with lead in mixed lead-zinc ores, which may be sulfidic or oxidized. The flotation process has provided an important tool for economically separating the lead from the zinc in the course of recovering these two valuable metals from mixed sulfide ores as it has been possible to use flotation to produce sulfide concentrates from both lead and zinc minerals. However, the flotation process has been an effective approach in separating lead from zinc in the treatment of oxidized lead-zinc ores [2-6].

Flotation of lead and zinc oxide minerals is more difficult than that of the corresponding sulfide minerals. The most common flotation technique commercially used for the treatment of lead and zinc oxide minerals is sulfidization with sodium sulfide followed by treatment with conventional cationic collectors such as amines [5, 7-10]. The consumption of collector may also be reduced by addingNa<sub>2</sub>S for sulfidization, which would change the mineral surface, and carbonate minerals accordingly become less hydrophilic [6, 11-13]. Adding an extra sulfide causes the lead and zinc oxide minerals to be depressed as it increases the negative charge that prevents the adsorption of collectors [6, 7, 9, 11-17].

Moreover, de-sliming has been an essential technique in most zinc oxide flotation projects [2, 8, 11, 18, 19]. The effect of dispersants such as Na<sub>2</sub>SiO<sub>3</sub> rather than de-sliming has been applied to decrease the effect of a large amount of slimes and fines [11].

It is mostly evident that the flotation methods [direct (first lead separation, then zinc), reverse (first zinc separation, then lead), and cumulative flotation of smithsonite and cerussite, then separation of two minerals] and the influential parameters such as the type of depressant and collectors and their dosages, and dosages of sodium sulfide and dispersant are the major factors affecting the flotation of smithsonite and cerussite, specially high calcareous/dolomitic Zn and Pb oxide ores. Therefore, the effects of these two main factors (flotation methods and influential parameters) would be comprehensively discussed in this paper. In this work, flotation of the Zn and Pb oxide minerals smithsonite and cerussite in the presence of carbonate gangue minerals was comprehensively investigated. Two-stage separation by the direct, reverse, and cumulative flotation approaches were evaluated using the anionic, cationic, and mixed collectors. A variety of influential parameters affecting the grade and recovery of the Zn and Pb concentrates were also optimized.

2. Material and methods

# 2.1. Ore sample and reagents

The representative sample used in this work was prepared from the Darreh-Zanjir mine located in the Yazd province (Iran). The XRF analysis (Philips Magix-Pro) of the ore sample is presented in Table 1. It indicates that the sample contains 23.39% CaO, 14.02% ZnO, 13.52% MgO, 3.24% PbO, and 2.86% Fe<sub>2</sub>O<sub>3</sub>, as the major elements,

and minor amounts of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. According to the XRD analysis (Bruker D8-advance diffractometer with Ni-filtered Cu-Ka radiation) and the optical mineralogy study, this sample includes smithsonite and hydrozincite as the zinc minerals; cerussite as the lead mineral; and also dolomite, calcite, and some other minerals. The optical mineralogical study of the thin and polished sections of screen size fractions indicated that about 80% of smithsonite and cerussite in size class of -100 µm was liberated. In addition, according to the results of the related articles, pH 10.5-11.5 was considered as the optimum pH range for concentrating the lead and zinc oxide minerals [7, 20-23].

The optical mineralogy study of the original sample was also performed; an image of this study is presented in Figure 1. The results obtained showed that the carbonate minerals and calcite are the dominant minerals in the feed sample. The lead and zinc minerals are present in the carbonate form.

In this work, Armac C (Cocoamine acetate), SIPX (Sodium Isopropyl Xanthate), and Aeroflot 3477 (sodium di-isobutyl dithiophosphate) as the collectors for zinc, KAX (potassium amyl xanthate) as the collector for zinc and lead, sodium sulfide as the activator for smithsonite and cerussite, sodium silicate as the dispersant, potassium dichromate as the depressant for lead, zinc sulfate as the depressant for zinc, MIBC (Methyl Isobutyl Carbinol) as the frother for lead, and pine oil as the frother for zinc were used. In most published articles, MIBC and pine oil have been used as the frother for lead and zinc enrichment, respectively [7, 23-26]. The reagents used in this investigation are listed in Table 2.

Component	CaO	ZnO	MgO	PbO	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	$Al_2O_3$	Cd	K <sub>2</sub> O	Na <sub>2</sub> O	L.O.
Amount (%)	23.39	14.02	13.52	3.24	2.86	0.85	0.16	0.15	0.06	0.04	40.9
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Table 1. XRF analysis of the representative sample.

Figure 1. An image from optic mineralogy study of the feed sample (Op and Cb: Zn-Pb oxide minerals; Cal: Calcite).

	Table 2. Reagents us	cu in the notation	i experiments.	
	Chemicals	Concentration	Supplier	Role
Armac C	Cocoamine acetate	99	Akzo Nobel	Collector for zinc
KAX	Potassium Amyl Xanthate	90	Cheminova	Collector for lead and zinc
SIPX	Sodium Isopropyl Xanthate	90	Cheminova	Collector for zinc
Aeroflot 3477	Sodium di-Isobutyl Dithiophosphate	-	Cytec	Collector for zinc
Sodium sulfide	Na <sub>2</sub> S	75	Merck	Sulfidizing reagent for lead and zinc
Copper sulfate	$CuSO_4$	97	Merck	Activator for zinc
Sodium silicate	Na <sub>2</sub> SiO <sub>3</sub>	97	Merck	Dispersant
Potassium dichromate	$K_2Cr_2O_7$	99	Scharlau	Depressant for lead
Zinc sulfate	ZnSO <sub>4</sub>	99	Merck	Depressant for zinc
Pine oil	Mixture	99	Penn Chemical	Frother for zinc
MIBC	Methyl Isobutyl Carbinol	99	Arkema	Frother for lead
Calcium oxide (lime)	CaO	99	Sigma-Aldrich	pH adjuster

Table 2. Reagents used in the flotation experiments.

#### 2.2. Flotation procedures

Flotation experiments were performed in a 2-L Denver cell running at 1100 rpm using 600 g of ore sample (30% pulp density). In the flotation tests in the laboratory scale, the effects of different parameters such as the amount of chemicals were investigated in the rougher and cleaner stages for separation of lead- and zinc-bearing oxide minerals in various flotation experiments (Figures 2a-e). Three methods including the direct, reverse, and cumulative flotations were tested as presented in Figures 2a-c. The influence of de-sliming prior to flotation of both lead and zinc minerals was evaluated, as given in Figure 2d. Finally, cleaning after the rougher stage was applied to enhance the grade of concentrate (Figure 2e). All the final experiments were performed in duplicate, and their average values were reported.



Figure 2. Five modes of flotation with relevant chemicals: a) direct flotation, b) reverse flotation, c) cumulative flotation, d) de-sliming prior to test, e) cleaning after rougher stage.

# 2.3. Flotation kinetic models

In this work, four flotation kinetic models adapted from other researchers [18] were selected to study the flotation performance in different stages for Zn and Pb oxide minerals, as shown in Table 3. The accumulative recoveries after 0.5, 1, 3, 5, 7, and 10 minutes of flotation were fitted to the four kinetic models. The statistical analysis software (SPSS) was used to simulate the flotation rate constant (k), maximum recovery  $(R_{\infty})$ , and correlation coefficient  $(R^2)$ . This calculation was presented for confirmation of the results, which showed that some kinetic models could correctly predict and investigate the behavior of zinc and lead oxide minerals in the flotation process.

Table 3. Desc	ription of the four	flotation kinetic	models used ir	ı this paper	[18].
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No.	Model	Formula	Remarks								
1	Classical first-order model	$R = R_{\infty}[1 - \exp(-k_1 t)]$	This model has been reported to predict values best when the recovery is especially low.								
2	First-order model with rectangular distribution of flotabilities	$R = R_{\infty} \{1 - \frac{1}{k_2 t} [1 - exp(-k_2 t)]\}$	This model has been reported to be the best of all models tested to fit experimental data because the rectangular distribution of flotabilities gives an added flexibility.								
3	Fully mixed reactor model	$R = R_{\infty}(1 - \frac{1}{1 + t/k_3})$	This model assumes that flotation components are exponentially distributed, which gives an added flexibility over the classical first-order model and enables it to fit the observed values very well.								
4	Improved gas/solid adsorption model	$R=R_{\infty}(\frac{k_4t}{1+k_4t})$	This mathematical form of model 4 is similar to model 3, which can be derived from model 3 by assuming $k_3 = 1 / k_4$ .								
	R = fractional recovery at time t; $R_{\infty}$ = fractional ultimate recovery; $k_n$ = rate constants (n = 1,, 4)										

# 3. Results and discussion

According to the results of the related articles, pH 10.5-11.5 was considered as the optimum pH range for concentrating the lead and zinc oxide minerals [7, 20-23]. The pH of pulp was adjusted with 1000 g/t CaO, which was added at the beginning of each experiment. CaO was used because it did not react with minerals in this range of pH [23, 24].

# **3.1.** Effects of direct and reverse flotation methods with and without depressants

Several tests were carried out to obtain the best flotation method for separation of these two oxide minerals. The chemicals used constantly in the experiments to concentrate the lead and zinc minerals were 1000 g/t of CaO to adjust the pH; 1000 g/t of sodium silicate in the separation of lead, and 200 g/t in the separation of zinc as dispersant; 2000 g/t of sodium sulfide as activator for both cerussite and smithsonite; 100 g/t of potassium amyl xanthate as a convenient collector for both minerals; and 50 g/t of MIBC and pine oil as frothers for lead and zinc, respectively.

Most lead minerals are floated easier than zinc minerals. Therefore, in most articles related to lead and zinc flotation, lead minerals have been floated at first (direct) regardless of the feed grade [1, 2, 6, 12, 15].

In the direct flotation approach, zinc sulfate (ZnSO<sub>4</sub>) with amounts of 300, 500, and 700 g/t was used to depress smithsonite in the lead flotation step. In addition, copper sulfate (CuSO<sub>4</sub>) with amounts of 500, 700, and 1000 g/t was employed to activate it in the next zinc flotation step [6]. On the other hand, in the reverse flotation approach, potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) with amounts of 200, 300, and 400 g/t was applied to depress cerussite in the zinc flotation step [14]. In addition, for a comparative purpose, both the direct and reverse flotation tests were carried out individually without adding any depressant but in the direct flotation tests without depressant, 200 g/t of CuSO<sub>4</sub> as an activator was also used constantly.

The results in Table 4 reveal that the direct flotation approach without any depressant yield the best results (Pb: grade=20.13% and recovery=60.7%; Zn: grade=18.65%; and recovery=54.02%). Therefore, the direct flotation method without any depressant was selected to be applied in the first processing step and their concentrates to be used for further stages to optimize the other influential parameters because the depressant of lead minerals depressed some zinc minerals as well, and vice versa.

Table 4. Direct and reverse notation approaches with and without depressants.											
			<b>K</b> <sub>2</sub>	$Cr_2O_7$ (g	g/t)	ZnS	O <sub>4</sub> /CuSO <sub>4</sub>	(g/t)	Without		
			200	300	400	300/500	500/700	700/1000	depressant		
	C reads $(0/)$	Zn	7.71	8.8	9.34	-	-	-	11.11		
Reverse flotation	Grade (%)	Pb	14.14	14.29	13.12	-	-	-	12.66		
	Recovery	Zn	21.88	21.03	23.05	-	-	-	44.43		
	(%)	Pb	25.02	22.02	23.92	-	-	-	21.98		
	C reads $(0/)$	Pb	-	-	-	21.91	20.26	18.92	20.13		
Direct flatation	Grade (%)	Zn	-	-	-	10.11	9.56	9.21	18.65		
Direct flotation -	Recovery	Pb	-	-	-	48.22	51.08	53.31	60.70		
	(%)	Zn	-	-	-	23.20	21.25	15.21	54.02		
	(%)	r o Zn	-	-	-	48.22 23.20	21.25	15.21	54.02		

Table 4. Direct and reverse flotation approaches with and without depressants.

# **3.2.** Effects of chemical dosages on lead flotation in rougher stage

According to the previous studies on cerussite flotation reagents and also some initial tests, the convenient chemical reagents and their dosages were considered as sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) as a dispersant in the range of 100-300 g/t [16, 23], sodium sulfide (Na<sub>2</sub>S) as an activator in the range of 2000-3000 g/t [2, 20, 25], and potassium amyl xanthate (KAX) as a collector in the range of 50-150 g/t [2, 4, 25]. In addition, 1000 g/t CaO to adjust pH and 50 g/t MIBC as a frother were constantly employed in all experiments [26].

### 3.2.1. Effect of Na<sub>2</sub>SiO<sub>3</sub> dosage

To find the optimum amount of sodium silicate as a dispersant, the three dosages 100, 200, and 300 g/t were selected. In these experiments, two other parameters (potassium amyl xanthate and sodium sulfide) were 100 g/t and 2500 g/t, respectively. The results of the first three experiments shown in Figure 3a yielded that the optimum dosage of Na<sub>2</sub>SiO<sub>3</sub> was 200 g/t with 34.62% Pb grade and 53.12% Pb recovery.

### 3.2.2. Effect of KAX dosage

In the second step, the collector dosage for cerussite flotation was optimized. Three tests were carried out with 50, 100, and 150 g/t of potassium amyl xanthate. The amounts of sodium silicate and sodium sulfide were selected as 200 g/t and 2500 g/t, respectively. The results of different experiments are presented in Figure 3b. It was concluded that the optimum dosage of collector was 150 g/t. The Pb grade and recovery in this step were 32.26% and 71.25%, respectively. In these series of tests, Pb grades did not show a great discrepancy but recovery increased drastically at the third level of collector dosage, which was a more important criterion in the rougher stage.

### 3.2.3. Effect of Na<sub>2</sub>S dosage

Finally, to optimize the amount of activator dosage in the cerussite flotation, the three different dosages of 2000, 2500, and 3000 g/t of sodium sulfide were applied. Two other parameters were set as 150 g/t for collector and 200 g/t for dispersant, which were determined in the previous steps. According to the results obtained (Figure 3c), 2500 g/t for activator dosage yielded the best Pb grade and recovery (32.26% and 71.25%, respectively).



Figure 3. Effects of chemicals dosages on lead rougher flotation: a) Effect of Na<sub>2</sub>SiO<sub>3</sub> concentration (KAX 100 g/t and Na<sub>2</sub>S 2500 g/t), b) Effect of KAX concentration (Na<sub>2</sub>SiO<sub>3</sub> 200 g/t and Na<sub>2</sub>S 2500 g/t), c) Effect of Na<sub>2</sub>S concentration (Na<sub>2</sub>SiO<sub>3</sub> 200 g/t and KAX 150 g/t).



Figure 3. Continued.

In the best conditions (Na<sub>2</sub>SiO<sub>3</sub>: 200g/t, KAX: 150g/t, and Na<sub>2</sub>S: 2500g/t), the concentrate with the lead content of 32.26% and the recovery of 71.25% and also the tail with the lead grade of 0.40% and the recovery of 28.75% were obtained. In addition, there was 13.51% Zn with the recovery of 96.88% in the tail, which entered the zinc separation stage. It was shown that lead flotation preceded by zinc flotation [2, 27].

# **3.3. Effects of chemicals dosages on zinc flotation in rougher stage**

According to the literature reviews on the smithsonite flotation and some preliminary tests, the convenient chemical reagents and their dosages were selected as sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) as a dispersant in the range of 1000-3000 g/t [16, 23, 28], sodium sulfide (Na<sub>2</sub>S) as an activator in the range of 2000-3000 g/t [2, 7, 10, 22, 23, 29], and potassium amyl xanthate with Armac C as a mixed collector in the range of 50-150 g/t [2, 9-11, 23, 30, 31]. In addition, 50 g/t pine oil was used as a frother constantly in all experiments [6, 8, 11, 32].

# **3.3.1. Effects of collectors types (Synergistic effect)**

The previous part of this work indicated that the best collector for flotation of cerussite was potassium amyl xanthate (KAX). Different collectors for flotation of smithsonite (ZnCO<sub>3</sub>) such as potassium amyl xanthate, Armac C, and sodium isopropyl xanthate (SIPX) were examined. In addition, a co-collector such as Aeroflot 3477 (sodium dibutyl dithiophosphate) was employed, and the synergistic effects of mixed collectors were studied [6].

The results presented in Table 5 show that the best Zn grade and recovery (29.73% and 49.67%, respectively) were achieved when the mixed

collectors including Armac C and KAX were used. The maximum recovery (73.21%) was achieved when Armac C was merely tested. In this case, Zn grade was similar to the feed content, which was not acceptable for this stage. It seems that the synergistic effect of mixed collectors would cause that Zn grade and recovery increase proportionally, and reach the optimum values. However, all tests were carried out using 100 g/t as collector/collectors. Since 50 g/t of each collector was used in test No. 3, the recovery of this test was lower than the other experiments. In order to find the optimum dosages for the two collectors (Armac C:A and KAX:K), nine experiments in combined form with three concentration values (50, 100, and 150 g/t) for

concentration values (50, 100, and 150 g/t) for each collector were examined (50A+50K, 50A+100K, 50A+150K, 100A+50K, 100A+100K, 100A+150K, 150A+50K, 150A+100K, and 150A+150K). Two other parameters, sodium silicate and sodium sulfide, were 2000 g/t and 2500 g/t, respectively. Based on the results of these nine experiments (Figure 5a), mixed collectors (Armac C+ KAX) with concentration of 150 g/t for each collector resulted in the optimal Zn grade and recovery for concentrate as 27.77% and 75.34%, respectively. At this stage, the recovery was more important than the grade, and in the last test (150A+150K), the recovery reached its maximum value. The grade was fluctuated in a limited range, and it was approximately constant for all the nine experiments. In addition, tests were performed (supplementary tests) at values higher than 150 g/t for collectors but no proper results were observed.

In the flotation of lead and zinc oxide minerals by mixed collectors, adding cationic/anionic collectors together [9, 11], Armac C and KAX increased the hydrophobicity by co-adsorption in the form of Armac C-KAX complex. At first, the surfaces of smithsonite and cerussite changed to ZnS and PbS by sulfidization with Na<sub>2</sub>S. Then KAX was adsorbed on ZnS(S<sup>-</sup>) and PbS(S<sup>-</sup>) by a negative-positive charge. In zinc oxide flotation with a mixed collector, Armac C may adsorb "strong spots", defined as the sites that are fully sulfidized and have a fairly high negative charge, covering the major areas of the smithsonite surface [11]. In addition, KAX adsorbed under the

above conditions may be adsorbed on the Armac C's positively charged head sites, depending on the sequence of addition (Figure 4). By using an appropriate concentration of KAX, this could be accomplished but by applying an excess KAX concentration, the recovery of Zn increased and the grade of Zn decreased. This could probably be attributed to the hypothesis that after adsorption of Armac C, extra KAX may adsorb as a co-collector complex or (KAX)<sub>2</sub> [11].

Tost No	Collector/Collectors	Concentration $(\mathbf{g}')$	<b>Zn (%)</b>		
Test No.	Conector/Conectors	Concentration $(-/t)$	Content	Recovery	
1	KAX (anionic)	100	23.13	50.13	
2	Armac C (cationic)	100	12.11	73.21	
3	Armac C + KAX (cationic + anionic)	50 + 50	29.73	49.67	
4	KAX + Aeroflot 3477 (anionic + promoter)	90 + 10	11.29	60.01	
5	SIPX (anionic)	100	18.11	51.36	

Table 5.	Effects	of types	of	collectors o	n Zn	flotation	process.
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Figure 4. Adsorption mechanisms of mixed collectors on the lead/zinc oxide minerals [11].

# 3.3.2. Effect of Na<sub>2</sub>SiO<sub>3</sub>

In the next step, and to achieve the optimum amount of sodium silicate, three experiments were performed using 1000, 2000, and 3000 g/t dosages. In these experiments, two other parameters (mixed collectors (Armac C+KAX) and sodium sulfide) were 150 g/t for each collector and 2500 g/t, respectively. According to the results given in Figure 5b, the optimal dosage of Na<sub>2</sub>SiO<sub>3</sub> was 2000 g/t with the same results obtained in the previous stage because both grade and recovery increased in this dosage. On the other hand, by reducing or increasing the dosage, all parameters decreased.

# 3.3.3. Effect of Na<sub>2</sub>S

In order to optimize the amount of activator on the smithsonite flotation, the three dosages of 2000, 2500, and 3000 g/t of sodium sulfide were examined. Two other parameters were set as 150 g/t for each collector in mixed form and 2000 g/t for dispersant, which were determined in the previous steps. According to the results (Figure 5c), the value of 2500 g/t for dispersant dosage resulted in the best grade and recovery. In the best conditions, the concentrate with the zinc content of 27.77% and the recovery of 75.34% was achieved. Also Zn grade and recovery for tail were 4.62% and 18.62%, respectively. Since by decreasing the amount of Na<sub>2</sub>S the recovery decreased, and by increasing this amount, the grade of Zn decreased, therefore, 2500 g/t was considered as the optimal value.



Figure 5. Effects of chemicals on zinc rougher flotation: a) Effect of KAX and Armac C concentration (Na<sub>2</sub>SiO<sub>3</sub> 2000 g/t and Na<sub>2</sub>S 2500 g/t), b) Effect of Na<sub>2</sub>SiO<sub>3</sub> concentration (150 g/t KAX, 150 g/t Armac C and Na<sub>2</sub>S 2500 g/t), c) Effect of Na<sub>2</sub>S concentration (150 g/t KAX, 150 g/t Armac C and Na<sub>2</sub>SiO<sub>3</sub> 2000 g/t).

# 3.4. Pb-Zn cumulative flotation tests

After determining the appropriate dosages of chemicals in the flotation of cerussite and smithsonite, the other mode of flotation approach, as the main separation method, was then tested. In this mode. both minerals were floated cumulatively, and were then separated from each other in the next step. In this case, a sharp decline in the recovery of both zinc and lead to about 36% and 15% was observed. In addition, in the second smithsonite separation stage, was not well-depressed because its surface was activated in the previous step. The contents of lead in the zinc concentrate and zinc in the lead concentrate were considerably high. Therefore, these two minerals were not separated from each other perfectly in this mode of flotation.

# 3.5. Effect of de-sliming

A major problem in processing the zinc flotation tailings is the large amount of fines and slimes, which increased the reagent consumption, causing the so-called slime coating phenomenon, hindering selectivity, and, in some cases, rendering the process unfeasible. To preclude this, de-sliming would be utilized [11, 28, 32]. The hydrocyclone used for de-sliming was a laboratory hydrocyclone with a separation cut size of 15 microns.

To investigate the effect of de-sliming on the flotation efficiency, two tests with de-slimed samples were carried out. De-sliming of sample was performed by hydrocyclone, and particles with size fraction of -20  $\mu$ m were separated. These two tests consisted of:

1. Feed de-sliming prior to separating cerussite and for the whole sample.

2. Pb tailing (first step) de-sliming prior to the flotation of smithsonite.

The results obtained are presented in Table 6, and in both cases of the de-sliming/flotation process, there is a decline in the grade and recovery of the Zn and Pb concentrates. Therefore, none of the two modes of de-sliming were conducted in the next step [11, 32]. In the first case of de-sliming, fines had 13.42% Zn (8 wt.%), and in the second case, fines had 16.73% Zn (10 wt.%).

Conditions		ead	Zinc			
Conditions	Content (%)	Recovery (%)	Content (%)	Recovery (%)		
Without de-sliming (-20µ)	32.26	71.25	27.77	75.34		
De-sliming before lead separation	16.27	47.03	20.92	68.24		
<b>De-sliming before zinc separation</b>	15.18	61.69	26.32	61.01		

 Table 6. Effect of de-sliming on content and recovery of lead and zinc.

# **3.6. Effects of chemical dosages on lead flotation in cleaner stage**

The cleaner stage was conducted in order to increase the Pb grade in the lead concentrate. In this case, the zinc-bearing minerals that remained in the lead concentrate were depressed by zinc sulfate ( $ZnSO_4$ ), and cerussite was reactivated by sodium sulfide ( $Na_2S$ ). It was re-floated by applying potassium amyl xanthate and MIBC as a collector and a frother.

### 3.6.1. Effects of ZnSO<sub>4</sub>, Na<sub>2</sub>S, and KAX

It should be noted that the pH of pulp containing lead concentrate was in the range of 10.5-11.5,

and therefore, there was no need to adjust the pH. At this stage, and to re-float the lead-bearing minerals, 50 g/t MIBC was used as a frother, and other influential chemicals were tested at two levels (ZnSO<sub>4</sub>: 500 and 1000 g/t; Na<sub>2</sub>S: 500 and 1000 g/t; and KAX: 50 and 100 g/t).

Figure 6 presents all the performed experiments and the optimal dosages of these three chemicals that were determined. The best result was obtained by 1000 g/t, 1000 g/t, and 50 g/t for  $ZnSO_4$ , Na<sub>2</sub>S, and KAX, respectively, with the best grade (49.82%) and recovery (60.06%).



Figure 6. Effects of chemical dosages on lead cleaner flotation: a) Effect of ZnSO<sub>4</sub> concentration (Na<sub>2</sub>S 1000 g/t and KAX 100 g/t), b) Effect of Na<sub>2</sub>S concentration (ZnSO<sub>4</sub> 1000 g/t and KAX 100 g/t), c) Effect of KAX concentration (ZnSO<sub>4</sub> 1000 g/t and Na<sub>2</sub>S 1000 g/t).

# **3.7. Effects of chemical dosages on zinc flotation in cleaner stage**

The cleaner stage was conducted to increase the Zn grade in the zinc concentrate. In this case, lead-bearing minerals and dolomite that remained in the zinc concentrate were depressed by potassium dichromate ( $K_2Cr_2O_7$ ), and also sodium silicate ( $Na_2SiO_3$ ) was used as a dispersant. Smithsonite was reactivated by sodium sulfide ( $Na_2S$ ) and it was re-floated with potassium amyl xanthate and Armac C as mixed collectors and also pine oil as a frother.

# 3.7.1. Effects of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, Na<sub>2</sub>SiO<sub>3</sub>, Na<sub>2</sub>S, KAX, and Armac C

At this stage, and to re-float the zinc-bearing minerals, 50 g/t pine oil was used as a frother, and other effective chemicals were tested at two levels ( $K_2Cr_2O_7$ : 0 and 50 g/t, Na<sub>2</sub>SiO<sub>3</sub>: 500 and 1000 g/t, Na<sub>2</sub>S: 500 and 1000 g/t, and KAX: 50 and 100 g/t + Armac C: 50 and 100 g/t). The pH of pulp was in the range of 10.5-11.5, and therefore, there was no need to adjust the pH.

Figure 7 illustrates the whole performed experiments and the optimal dosages of the five chemicals that were achieved. The best result was obtained by 0, 500 g/t and 1000 g/t for  $K_2Cr_2O_7$ ,  $Na_2SiO_3$ , and  $Na_2S$ , respectively, and 50 g/t for each collector (KAX and Armac C). The best grade and recovery were 35.47% and 68.56%, respectively.



Figure 7. Effects of chemical dosages on zinc cleaner flotation a) Effect of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> concentration (Na<sub>2</sub>SiO<sub>3</sub> 500 g/t, Na<sub>2</sub>S 500 g/t, KAX 50 g/t, and Armac C 50 g/t), b) Effect of Na<sub>2</sub>SiO<sub>3</sub> concentration (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> not used, Na<sub>2</sub>S 500 g/t, KAX 50 g/t, and Armac C 50 g/t) (c) Effect of KAX and Armac C concentration (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> not used, Na<sub>2</sub>SiO<sub>3</sub> 500 g/t, and Na<sub>2</sub>S 500 g/t), d) Effect of Na<sub>2</sub>S concentration (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> not used, Na<sub>2</sub>SiO<sub>3</sub> 500 g/t, and Na<sub>2</sub>S 500 g/t), d) Effect of Na<sub>2</sub>S concentration (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> not used, Na<sub>2</sub>SiO<sub>3</sub> 500 g/t, and Armac C 50 g/t).

# 3.8. Kinetic study

Kinetic models of flotation have been widely applied based on an analogy with the homogeneous chemical kinetics [33]. Flotation kinetic tests were carried out to confirm the results obtained at each steps (zinc and lead in the rougher and cleaner stages). Based on the 4 models mentioned in the materials and methods section, all results are presented in Tables 8a, 8b, 9a, and 9b. It is clear in Figures 8 and 9 that all model analyses confirm the accuracy of the results. This kinetic study was presented only for confirmation of the results. In the lead rougher stage, models 1-4 (with k = 0.44, 0.85, 1.09, and 2.28 and R = 76.53, 87.01, 100, and 95.72%), and in the zinc rougher stage, models 1-4 (with k = 0.36, 0.93, 0.98, and 2.1 and R = 78.68, 100, 88.91, and 97.27%), respectively, had the best

fitting with the original data. Also in the lead cleaner stage, models 1-4 (with k = 0.36, 0.67, 1.02, and 1.7 and R = 100, 100, 100, and 100%), and in the zinc cleaner stage, models 1-4 (with k = 0.46, 0.92, 1.23, and 2.07 and R = 98.44, 100, 100, and 100%), respectively, had the best fitting with the original data.

 Table 7a. Non-linear regression results for all models fitting to the experimental data for rougher stage of lead flotation.

Time	Exposimontal	E	q. (1) (Model	1)	E	q. (2) (Model	2)	E	q. (3) (Model	3)	E	q. (4) (Model	4)
(min)	CR (%)	CR (%)	Deviation	Dev.^2									
0	0	0	0	0	0	0	0	0	0	0	0	0	0
0.5	15.22	14.99	0.229	0.053	16.08	-0.857	0.735	17.23	-2.004	4.017	19.96	-4.741	22.472
1	31.21	27.05	4.155	17.267	28.33	2.879	8.288	29.20	2.006	4.025	32.03	-0.823	0.677
3	53.37	55.85	-2.482	6.161	55.47	-2.107	4.437	54.41	-1.042	1.086	55.04	-1.669	2.784
5	65.91	67.88	-1.978	3.911	66.77	-0.860	0.740	65.76	0.145	0.021	65.12	0.783	0.614
7	73.33	72.92	0.412	0.170	72.38	0.950	0.903	72.22	1.107	1.227	71.02	2.306	5.316
10	77.23	75.55	1.681	2.826	76.74	0.492	0.242	77.96	-0.730	0.533	76.50	0.738	0.544
K		0.44			0.85			2.28			1.09		
R		76.53			87.01			95.72			100		
DEVS		2.02			0.50			-0.52			-7.15		
DEVSQ		30.39			15.35			10.91			34.40		
R <sup>2</sup>		0.99			0.995			0.996			0.989		

 Table 7 b. Non-linear regression results for all models fitting to the experimental data for rougher stage of zinc flotation.

	notation												
T:	F	E	Eq. (1) (Model 1)			q. (2) (Model	2)	E	q. (3) (Model	3)	E	q. (4) (Model	4)
(min)	CR (%)	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2
0	0	0	0	0	0	0	0	0	0	0	0	0	0
0.5	18.13	12.84	5.290	27.991	18.72	-0.59	0.347	18.73	-0.60	0.360	17.94	0.19	0.036
1	33.40	23.58	9.819	96.414	32.38	1.02	1.047	31.41	1.99	3.969	29.38	4.02	16.179
3	55.24	51.66	3.580	12.821	60.41	-5.17	26.747	57.25	-2.01	4.041	52.34	2.91	8.449
5	68.24	65.43	2.809	7.893	71.01	-2.77	7.649	68.53	-0.29	0.083	62.84	5.40	29.143
7	76.11	72.18	3.926	15.415	76.04	0.07	0.005	74.85	1.26	1.596	69.10	7.01	49.107
10	80.27	76.45	3.818	14.580	79.89	0.38	0.141	80.41	-0.14	0.019	74.99	5.28	27.90
K		0.36			0.985			2.1			0.93		
R		78.68			88.91			97.27			100		
DEVS		29.25			-7.059			0.221			24.807		
DEVSQ		175.12			35.935			10.066			130.81		
R <sup>2</sup>		0.952			0.988			0.997			0.958		



Figure 8. Comparison of different kinetic models fitted to the test data (A) Lead (B) Zinc in rougher stage.

	ilotation.												
Time	Exposimontal	I	Eq. (1) (Model	1)	E	q. (2) (Model	2)	Eq. (3) (Model 3)			Eq. (4) (Model 4)		
(min)	CR (%)	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2
0	0	0	0	0	0	0	0	0	0	0	0	0	0
0.5	13.76	16.32	-2.56	6.531	15.06	-1.299	1.688	22.66	-8.894	79.11	19.16	-5.40	29.16
1	36.43	29.97	6.463	41.78	27.17	9.259	85.73	36.94	-0.507	0.257	31.02	5.410	29.26
3	63.77	65.66	-1.877	3.525	56.99	6.790	46.11	63.74	0.045	0.002	54.16	9.618	92.51
5	82.23	83.16	-0.926	0.857	71.26	10.972	120.38	74.55	7.683	59.03	64.50	17.73	314.43
7	92.38	91.74	0.644	0.415	78.92	13.46	181.17	80.40	11.99	143.7	70.59	21.79	474.82
10	99.19	97.16	2.027	4.107	85.13	14.06	197.68	85.42	13.77	189.6	76.28	22.91	524.83
K		0.36			0.677			1.707			1.018		
R		100			100			100			100		
DEVS		3.775			53.24			24.09			72.059		
DEVSQ		57.21			632.76			471.79			1465		
$\mathbb{R}^2$		0.990			0.888			0.917			0.741		

Table 8 a. Non-linear regression results for all models fitting to the experimental data for cleaner stage of lead flotation

Table 8 b. Non-linear regression results for all models fitting to the experimental data for cleaner stage of zinc

notation.														
Time	Fynarimantal	ŀ	Eq. (1) (Model	1)	E	q. (2) (Model	2)	Eq. (3) (Model 3)			E	Eq. (4) (Model 4)		
(min)	CR (%)	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2	CR (%)	Deviation	Dev.^2	
0	0	0	0	0	0	0	0	0	0	0	0	0	0	
0.5	19.98	20.52	-0.541	0.293	19.87	0.107	0.011	19.46	0.517	0.267	22.12	-2.141	4.584	
1	43.76	36.76	6.991	48.88	34.66	9.090	82.63	32.58	11.171	124.79	34.87	8.880	78.86	
3	70.04	74.23	-4.193	17.59	66.11	3.929	15.44	59.18	10.852	117.78	58.20	11.84	140.17	
5	85.46	88.94	-3.481	12.12	78.51	6.942	48.20	70.73	14.725	216.81	68.08	17.37	301.81	
7	96.14	94.71	1.432	2.049	84.52	11.62	135.0	77.19	18.957	359.36	73.78	22.36	500.11	
10	99.98	97.53	2.455	6.026	89.15	10.83	117.3	82.86	17.123	293.18	79.01	20.97	439.60	
K		0.467			0.921			2.069			1.23			
R		98.44			100			100			100			
DEVS		2.661			42.52			73.34			79.28			
DEVSQ		86.95			398.61			1112.2			14.65.1			
$\mathbb{R}^2$		0.983			0.920			0.778			0.707			



Figure 9. Comparison of different kinetic models fitted to the test data (A) Lead (B) Zinc in cleaner stage.

# **3.9.** Characterization studies

# 3.9.1. XRD and XRF analyses

The results of XRF analysis of the zinc and lead concentrates are presented in Table 9. The lead concentrate sample contained 50.84% Pb and the zinc concentrate sample contained 31.98% Zn. However, the XRD analysis and the optical mineralogy performed on the zinc concentrate

sample indicated the following minerals: smithsonite, dolomite, hydrozincite, and calcite. In addition, in the lead concentrate sample, there were cerussite, dolomite, hydrozincite, calcite, and smithsonite. These analyses showed that in both concentrates, there were considerable amounts of Mg and Ca, which is an indication of the existence of dolomite in the concentrates.

Table 9. ARF analysis of leau and zine concentrates	Table 9. XRF	analysis	of lead	and zinc	concentrates
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Phase	Pb	SO <sub>3</sub>	CaO	Zn	MgO	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	MnO	Cu	BaO	$P_2O_5$
Lead conc. %	50.84	7.77	7.69	6.54	3.48	2.37	0.79	0.18	0.1	0.03	0.02
Phase	Zn	CaO	Pb	MgO	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	SO <sub>3</sub>	MnO	Cu	BaO	$P_2O_5$
Zinc conc. %	31.98	9.16	5.31	4.46	4.07	2.19	0.95	0.29	0.11	0.01	<

**3.9.2. SEM-EDS analysis of Pb-Zn concentrates** The morphology of the lead and zinc minerals in the concentrates were evaluated by SEM, and chemical analysis was performed by the EDS method, whose results are given in Figure 10. The EDS analysis presented that the Pb concentrate had the highest amounts of Pb (43.41%), C (20.05%), and O (14.42%). This indicates that the main mineral of the lead concentrate is cerussite with the PbCO<sub>3</sub> formula. In addition, for the zinc concentrate, the EDS analysis showed that the average contents of Zn, O, and C were 34.05%, 33.13%, and 16%, respectively. Also this data represents the fact that the major Zn-bearing mineral is smithsonite with the ZnCO<sub>3</sub> formula.



Figure 10. Scanning electron micrographs (SEM) and Energy Dispersive Spectroscopy (EDS): a) lead concentrate, b) zinc concentrate.

# 4. Conclusions

The following conclusions were made as a result of this experimental research work, in which a lead-zinc carbonate ore sample from Darreh-Zanjir mine located in the Yazd province (Iran) was used: - The representative sample with the lead grade of 2.5% and zinc grade of 9.39% contained cerussite (PbCO<sub>3</sub>) and smithsonite (ZnCO<sub>3</sub>) as the dominant Pb- and Zn-bearing oxide minerals.

- The results obtained indicated that the dosages of sodium sulfide and sodium silicate, and also the

type of collector were the effective factors on the recovery of smithsonite and cerussite. The optimization process was performed by changing the type of chemicals and their dosages. Desliming prior to the flotation and cumulative flotation tests were also investigated, which did not yield the desired results.

- The flotation results in the rougher stage showed that by using 1000 g/t lime with 10.5 < pH < 11.5, 200 g/t sodium silicate as the dispersant, 2500 g/t sodium sulfide as the activator of cerrusite, 150 g/t potassium amyl xanthate as the collector, and MIBC as the frother, the lead concentrate with Pb grade and recovery of 32.26% and 71.25%, respectively, was obtained. In addition, under the optimal conditions (10.5 < pH < 11.5, 2000 g/t sodium silicate as the dispersant, 2500 g/t sodium sulfide as the sulfidizing agent, 150 g/t potassium amyl xanthate with 150 g/t Armac C as the mixed collectors. and pine oil as the frother), the zinc concentrate with Zn grade and recovery of 27.77% and 75.34%, respectively, was obtained.

- The results obtained showed that after the cleaner stage for both lead and zinc, a cerussite concentrate with Pb grade and recovery of 49.82% and 60.06%, respectively, and a smithsonite concentrate with Zn grade and recovery of 35.47% and 68.56%, respectively, were obtained under the optimal conditions.

- After investigating the synergistic effects of the mixed collectors (Armac C + KAX), and also adsorption mechanisms of collectors on the minerals' surfaces, the results obtained showed that at a fixed amount of Armac C, when the KAX concentration decreased, the recovery increased and enhanced the amine flotation recovery.

- Kinetics of Zn and Pb oxide mineral flotations in the rougher and cleaner stages showed that some kinetics models, especially the classical first-order model, could predict the flotation behaviour of the Zn and Pb oxide minerals.

# Acknowledgments

This work was supported by the Kanifaravaran Company and the University of Tehran. Thus the authors are grateful to Mr. Sedehi and Mr. Koleini, the Head of Kanifaravaran Company. We would also like to thank our honorable partners on the project for their contributions to the work reported in this paper.

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# مطالعه مقایسهای در مورد فلوتاسیون دو مرحلهای کانیهای سرب و روی اکسیده با استفاده از کلکتورهای آنیونی، کاتیونی و مخلوط (کاتیونی و آنیونی)

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ارسال ۲۰۱۸/۲/۷ پذیرش ۲۰۱۸/۲/۷

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### چکیدہ:

نمونه خوراک سرب و روی کربناته شامل ۲/۱٪ سرب و ۹۳/۹٪ روی موضوع این پژوهش است. نمونه از معدن دره زنجیر واقع در استان یـزد در ایـران تهیـه شـده است. در این مطالعه، تأثیر فاکتورهای مؤثر بر فلوتاسیون اسمیت زونیت و سروزیت مورد بررسی قرار گرفته است. میان پارامترهای مختلف، مقـدار متفـرق کننـده، بازداشت کنندهها، عاملهای سولفید کننده و کلکتورها، نرمه گیری قبل از فلوتاسیون سرب و روی، پارامترهای مـؤثر بـر بازیـابی و عیـار کنسـانترههـای حاصـل از فلوتاسیون سرب و روی میباشند. علاوه بر آن، کلکتورهای آنیونی، کاتیونی و مخلوط (آنیونی و کاتیونی) برای فلوتاسیون سرب و روی به کار گرفتـه شـدند. نتـایج فلوتاسیون معکوس و تجمعی سرب و روی در مقایسه با فلوتاسیون مستقیم بدون استفاده از بازداشت کننده، نسبتاً کم بود که مورد بررسی قرار گرفتـه شـدند. نتـایج اسمیت زونیت با مخلوط کلکتورها (آرماک سی و امیل گزنتات پتاسیم) نتایج مناسبی را به همراه داشت. همینطور، مقادیر مـواد شـیمیایی در مرحلـه فلوتاسـیون شستشوی سرب و روی بهینهسازی شد و در نهایت کنسانتره سرب با عیار ۲۲/۲۶٪ و راندمان کننده، نسبتاً کم بود که مورد بررسی قرار گرفت. فلوتاسـیون دست آمد. علاوه بر این، سرحان می و امیل گزنتات پتاسیم) نتایج مناسبی را به همراه داشت. همین طور، مقادیر مـواد شـیمیایی در مرحلـه فلوتاسـیون دست آدم بر و روی بهینهسازی شد و در نهایت کنسانتره سرب با عیار ۲۲/۲۶٪ و راندمان ۲۱/۲۵٪ و کنسانتره روی با عیـار ۲۷/۷۲٪ و رانـدمان ۲۵/۲۷٪ بـه مستشوی سرب و روی بهینهسازی شد و در نهایت کنسانتره سرب با عیار ۲۲/۲۶٪ و راندمان ۲۱/۲۵٪ و کنسانتره روی با عیـار ۲۷/۷۱٪ و رانـدمان ۲۵/۲۷٪ بـه مستشوی سرب و روی بهینهسازی شد و در نهایت کنسانتره سرب با عیار ۲۲/۲۶٪ و راندمان ۲۱/۲۵٪ و کنسانتره روی با عیـار ۲۷/۷۱٪ و رانـدمان ۲۵/۲۷٪ بـه دست آمد. علاوه بر این، سینتیک فلوتاسیون کانیهای اکسیده سرب و روی در مراحل اولیه و شستشو مورد بررسی قرار گرفت که مدلهـای سینتیکی مخصوصاً

**کلمات کلیدی:** فلوتاسیون، کلکتورهای کاتیونی و آنیونی، کانیهای اکسیدی سرب و روی.