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Concentrating the Zn-Pb sample from Nignan ore deposit

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Preliminary communication

Maryam Abedi Moghadam[1], **Marzieh Hosseini Nasab**[2*], **Mohammad Noaparast**[1]

[1] *School of Mining Engineering, College of Engineering, University of Tehran, Tehran, Iran.*

[2] *Department of Mining Engineering, University of Sistan and Baluchestan, Zahedan, Iran.*

Abstract

The aim of the present study is to increase the grade of zinc from lead and zinc ore from the Noahang (Chah-Ahan) area of the Nignan deposit to more than 40% Zn, using the flotation method. The experiments were conducted on a low-grade sample with average grades of 3% zinc and 0.05% lead. The main zinc minerals in this sample were sphalerite and smithsonite in the carbonate field, and galena was rarely observed. The results of mineralogical studies and polished thin sections showed that the degree of liberation of zinc ores in the size fraction -106+75 µm was 90-95%. Different parameters such as size, solid mass percentage, type and dosage of collector were investigated, and under optimal conditions, a concentrate with a grade of 5% Zn and 70.4% recovery was obtained. In addition, kinetic experiments were conducted to achieve the highest Zn recovery in the rougher stage, which reached up to 58.6% of recovery. The results indicated that the best grade and recovery of zinc were achieved when the sample was crushed with a wet rod mill for 8 minutes to reach to the desired degree of liberation. The final grade and recovery of flotation method were obtained as 42.19 and 40.08% for zinc concentrate, respectively.

Keywords:
Noahang mineral area, flotation, degree of liberation, grade, recovery

1. Introduction

Metals have many applications in industries (**Hosseini Nasab and Rezazadeh, 2022; Hosseini Nasab, 2024**), and lead and zinc are among the most widely used non-ferrous metals in various industries. Oxidized ores of these two metals are the most important source of production of these metals after their sulfide types. Despite the problems and complexities in this field (due to the nature of these types of ores), the flotation method is the most common method for their processing (**Moradi, 2005**). Nowadays, due to the reduction of high-grade mineral reserves and the economic exploitation and processing of low-grade minerals, low-grade metal ores have become of particular importance (**Moradi, 2005; Maleki et al., 2023; Faramarz et al., 2024**). Therefore, the low-grade lead and zinc-oxidized ores are also considered among these reserves (**Moradi, 2005**).

Due to the similarity of genesis and formation of lead and zinc minerals, despite their different physical and chemical properties, they are mostly found together in mines (**Ghorbani, 2008**). The main use of lead metal is in transportation and electrical, and zinc is mainly used as part of battery tanks, in foundry molds and in the automotive industry. Zinc is also used in galvanizing steel products and prevents the corrosion of steel (**Goodwin, 2008**). Most zinc oxides and carbonate minerals have very fine crystal sizes (3-10 µm), and are available in carbonate, oxide and silicate forms (**Boni, 2003**). Lead is found in nature as sulfide, sulfate, carbonate and lead salts (**Black and Allen, 1999**). The most common methods for increasing the grade of lead and zinc ores include flotation and gravity methods in which gravity separation is less commonly used than flotation (**Karimpour and Saadat, 2006**). Mainly, process optimization can be pursued more effectively and with a lower percentage of error by correcting the type and amount of chemical materials used. In this regard, by conducting targeted experiments, more suitable chemicals that are employed on a commercial scale in the world would normally be used which yield the appropriate results, as well (**King, 2001**).

In the present study, the feasibility of flotation of lead and zinc ores from the Nignan deposit, which is located in South Khorasan Province, Iran, was investigated, and the process was optimized in terms of the type and amount of chemical materials used, including collectors, *depressants,* and activators. This research, using case studies and statistical analyses, seeks to find the optimal flotation conditions for zinc ore from the Nignan deposit

* Corresponding author: Marzieh Hosseini Nasab
e-mail address: hosseininasab@eng.usb.ac.ir
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that would lead to achieving the best possible grade and recovery.

1.1. Research objectives

Most of the extracted mineral soils containing lead and zinc are of the sulfide and oxide type, and therefore the technical processing approach which is accordingly employed is usually the flotation method. The objectives of the present research can be briefly stated in the following:

- study on the concentrating of lead and zinc ore sample from the Nignan deposit, using flotation.
- achieving a grade of more than 40% zinc, in zinc concentrate with acceptable recovery.

To achieve these objectives, it is necessary to organize and implement the appropriate flotation experiments on a laboratory scale for the ore sample of the Nignan mine. Flotation experiments were conducted using potassium amyl xanthate (PAX), coco alkyl amine acetate (Armac C) and their combination as collectors, zinc sulfate and copper sulfate as zinc *depressant* and activator, and polypropylene glycol (A65) as a frother.

Before conducting flotation experiments, sampling was performed and the samples were crushed. Then, a sample was subjected to sieve analysis and the distribution of lead and zinc metals in different size fractions were determined. The work index of the sample was also experimentally calculated. To determine the degree of liberation of the ore, thin polished sections were prepared to run the mineralogical and microscopic studies to use its results in the flotation process.

1.2. Introduction to the Nignan Complex

Due to the proximity of the Nignan lead and zinc exploration area to two important crustal blocks of Iran (the Lut and Tabas blocks), and the proximity to the Nayband fundamental fault, this part of central Iran has been affected by the geodynamic processes governing both crustal blocks, and numerous lead and zinc deposits have formed there. According to field observations and studies of polished sections, the mineralogy in this area is simple and consists of primary minerals sphalerite, pyrite and hematite, as well as secondary minerals cerussite, hemimorphite, smithsonite, goethite and limonite. Galena, calcite and dolomite are the main tailings, and quartz and barite are present in smaller amounts (**Gholami and Ebrahimi, 2010**).

1.3. Research background

In this section, while reviewing previous research on the related topic, the working method and chemicals used will be discussed.

Moradkhani et al. (2010) conducted a study on the effect of Armac-C collector on the flotation of non-sulfide ores in the presence of a KAX collector. In this study, the effect of cationic collector concentration (coco-alkyl-amine-acetate) on the optimization of zinc grade and recovery in flotation using combined collectors (KAX-Armac C) was investigated. The orthogonal array design L18 (21×37) was selected according to the Taguchi method, which includes one parameter with two levels and seven parameters with three levels. The parameters included the concentration of sodium silicate, starch, sodium sulfide, xanthate (in two stages) and Armac-C concentration, pH and flotation time. To determine the relationship between experimental conditions and recovery levels, statistical analysis and S/N evaluation methods were used. According to the obtained results, the Armac-C concentration had the greatest effect on zinc recovery in the process.

Mahdilou et al. (2013) conducted a study on the flotation of zinc oxide ore using cationic and cationic-anionic composite collectors. In this research, the flotation of smithsonite was investigated, using the cationic collectors Armac-C and Armac-T, and their mixture with potassium amyl xanthate (KAX) as an anionic collector. The results showed that the Armac-T collector was slightly more selective than Armac-C, but their combination with KAX acted in the opposite way. The KAX-Armac C composite collector improved the zinc grade and recovery of the flotation concentrate, while KAX-Armac T did not show any positive effect. At a KAX-Armac C mixture ratio of 1:2, the amine collector consumption was reduced, and an optimal concentrate with 94% recovery and 43% zinc grade was obtained without any depressing agent.

Mehrabani et al. (2010) conducted a study on the optimization and modelling of the flotation process of a low-grade lead and zinc ore (3% zinc and 1% lead). In this study, three parameters, activator (copper sulfate), collector (potassium amyl xanthate), and pH, were investigated at five levels. The RSM method was used to analyze the experiments and accordingly model the process. Analysis of variance indicated that the most important factor affecting the recovery and separation efficiency of zinc was the collector, and in the optimization of the process, the maximum recovery and separation efficiency values were 79.12% and 64.59%, respectively, using a concentration of 150 g/t of activator, 120 g/t of collector at a pH of 10.4. However, in optimization studies, a case was reported in which using the same reagents, the recovery and separation efficiency of zinc were improved by 9 and 5%, respectively.

Seke (2005) optimized the selective flotation of galena and sphalerite from the Pinah mine. This study was conducted to determine the flotation capability of galena and sphalerite when the sulfide sample was a mixture of lead, zinc, and copper. Since sphalerite recovery increased with galena recovery and increased concentrate volume, xanthate collector could not be used. However, by activating sphalerite with copper (II) ions, sphalerite recovery increased but galena recovery decreased. Due to the texture of the ore, cyanide alone was not capable of depressing sphalerite. The simultaneous use of cyanide and zinc sulfate improved sphalerite depressant compared to cyanide alone. To improve the selection of galena and sphalerite, the rougher lead concentrate must be re-ground because prior to this step, the separation of lead and zinc was poor.

Dehghani and Shahbazi (2017) conducted a study entitled "Depression of pyrite and sphalerite in the rougher flotation of galena from Gushfeel lead and zinc ore". The lead and zinc rich-pyrite ore of the Gushfeel underground mine in Irankooh has a complex mineralogical composition. The lead concentrate obtained from the flotation of this ore contains amounts of iron and zinc exceeding the permissible limits. In this study, the effect of zinc sulfate, sodium cyanide, and iron sulfate on the depressant of sphalerite and pyrite in the flotation of galena from the Gushfeel ore was investigated. Experiments were conducted on samples finer than 75 and 90 μm, and it was observed that during galena flotation in 75 μm samples and at normal pH, with increasing zinc sulfate consumption from 300 to 1000 g/t, the lead grade in the lead concentrate increased from 4.5 to 7.1%, while its recovery remained unchanged. On the other hand, the iron grade of the concentrate decreased from 13 to 10% and the recovery of iron and zinc decreased by about 6.5 and 7%, respectively. In flotation of 90 μm samples, with increasing zinc sulfate from 500 to 1000 g/t, the iron and lead grades increased slightly, but the zinc grade decreased. In this case, the recovery of lead, zinc and iron in the lead concentrate decreased by about 7, 4 and 2% respectively, and with a further increase in zinc sulfate, the recovery of zinc and iron in the lead concentrate increased. With an increase in sodium cyanide consumption from 100 to 200 g/t, the iron content in the lead concentrate did not change, but its lead and zinc content decreased by 2 and 0.4%. Factorial experiments conducted, at two levels of zinc sulfate, sodium cyanide and pH parameters, also showed that the effect of the parameters depends on the pH of the environment. In addition, the maximum recovery of lead in these conditions was about 30%, while the recovery of zinc and iron in the lead concentrate in this case was about 7.9% and 5.2%, respectively.

Mahdavi Amin et al. (2019) conducted a study entitled "Evaluation of the role of particle size in the performance of the lead and zinc flotation circuit of the Bama-Irankooh mining complex". In this research, the effect of particle size on flotation was investigated on two industrial and laboratory scales by dividing particles into three size ranges: coarse, medium, and fine. To increase recovery, the amount and distribution of chemicals changed. For this purpose, first, on a laboratory scale using the Taguchi 9L design, the effect of the amount and type of chemicals on recovery was investigated, and the results showed that using 30 g/t of Aero 3477 collector in the rougher cells along with 15 g/t of potassium amyl xanthate collector, and 7 g of MIBC frother in the scavenger cells increased the recovery of coarse particles by 5.4%. In addition, using 15 g/t of Aero 3477 in the rougher cells along with 7 g/t of potassium amyl xanthate and 30 g/t of MIBC frother in the scavenger cells would increase the recovery of fines by 5.3%. Finally, the best distributions obtained from the experiments were implemented in the plant and it was found that using 30 g/t of Aero 3477 collector in the rougher cells along with 15 g/t of potassium amyl xanthate collector and 7 g/t of MIBC frother in the scavenger cells increased the recovery of fines and coarse particles by 2.5% and 3.9%, respectively, and increased the grade of zinc concentrate by 4.1%.

The flotation of oxide minerals of lead and zinc, especially zinc oxides, is much more difficult than the flotation of sulfide minerals. Lead, zinc, and copper oxides are much more difficult to be floated due to their higher solubility and greater surface hydration compared to sulfides (**Fuerstenau et al., 1987, Irannejad et al., 2011**). Prior to the flotation of lead and zinc ores at the Irankooh (Bama) mines, the ore is pre-processed by the heavy medium gravity (HMS) method. The centrifugal separator in this unit is a Dyna Whirlpool (DWP) type, and this operation was performed after secondary crushing. The lead flotation process of the plant was as follows: the pulp overflowing from the cyclone was fed to two preparation units with sizes of <150 μm, which are placed in series. Sodium sulfide is added to the first preparation unit and potassium amyl xanthate is added to the second, and the frother is added to the pulp as it enters the first flotation cell. The chemicals used in lead flotation include sodium silicate (500 g/t), sodium sulfide (25.3 kg/t), potassium amyl xanthate (300 g/t), and a frother (250-400 g/t) (**Moradi, 2001**). The zinc flotation process is as follows: the fines were removed from the tailings of the lead circuit by two cyclones installed in series and then tailings were fed to the zinc preparation units. For the zinc circuit, sodium sulfide and sodium silicate were added in the first preparation, and Armac TD was added in the second *preparation*. The zinc circuit consists of 12 cells of 2 m^3; so that the product of the first 6 cells was removed as concentrate and the product of the second 6 cells was returned to the preparation stage (**Karam Soltani, 1999**).

2. Methods

2.1. Sample preparation

For the purpose of preliminary studies and feasibility study of the enrichment of the sample from the Nignan lead and zinc deposit, approximately 40 kg of crushed sample was sent to the Mineral Processing Laboratory of the University of Tehran. For laboratory studies, after homogenization and preparation of a representative sample, it was forwarded to the assay laboratory for feed chemical analysis, XRF, XRD, ICP analyses, and microscopic studies. After identifying the characteristics of the sample and determining the degree of liberation of valuable minerals, initial flotation experiments were designed to find the optimal grade and recovery.

Table 1. Feed analysis results

Sample Code	Zn_T (%)	Zn_O (%)	Pb_T (%)	Pb_O (%)	Cu_T (%)	Cu_O (%)	Ag (ppm)
Feed	3.01	1.66	0.05	0.02	nd*	nd*	5

*nd: Not Detected

Table 2. XRF Analysis Results

Sample	SiO_2	Al_2O_3	CaO	Fe_2O_3	ZnO
	%	%	%	%	%
	14.5	3.6	36.8	3.5	4
	SO_3	Na_2O	K_2O	TiO_2	La&Lu
	%	%	%	%	%
	2.5	0.5	0.5	0.2	<0.1
	PbO	L.O.I	MgO	Cl	P_2O_5
	%	%	%	%	%
	<0.1	31.3	2.6	<0.1	<0.1

2.2. Analyses of the feed

In order to check the lead and zinc content and analyze XRD, XRF and ICP, a representative sample was prepared as feed and sent to the desired laboratories. The results of the feed analysis are in **Table 1**.

As can be seen in **Table 1**, the prepared sample contains about 3% zinc, and has a low lead content, and

Table 3. XRD analysis results

Sample XRD		
Lamp type: Cu	Voltage: 40KV	Current: 30mA
Nsteps: 200	Step Size: 0/01	2θ: 5-90
Name	**Formula**	
Calcite	$CaCO_3$	
Quartz	SiO_2	
Ankerite	$Ca_{0.997}(Mg_{0.273}Fe_{0.676}Mn_{0.054})(CO_3)_2$	
Smithsonite	$ZnCO_3$	
Kaolinite	$Al_2Si_2O_5(OH)_4$	
Glaucophane	$Na_2(Mg,Fe,Al)_5Si_8O_{22}(OH)_2$	
Sphalerite	ZnS	
Gypsum	$CaSO_4.2H_2O$	
Illite	$KAl_2Si_3AlO_{10}(OH)_2$	

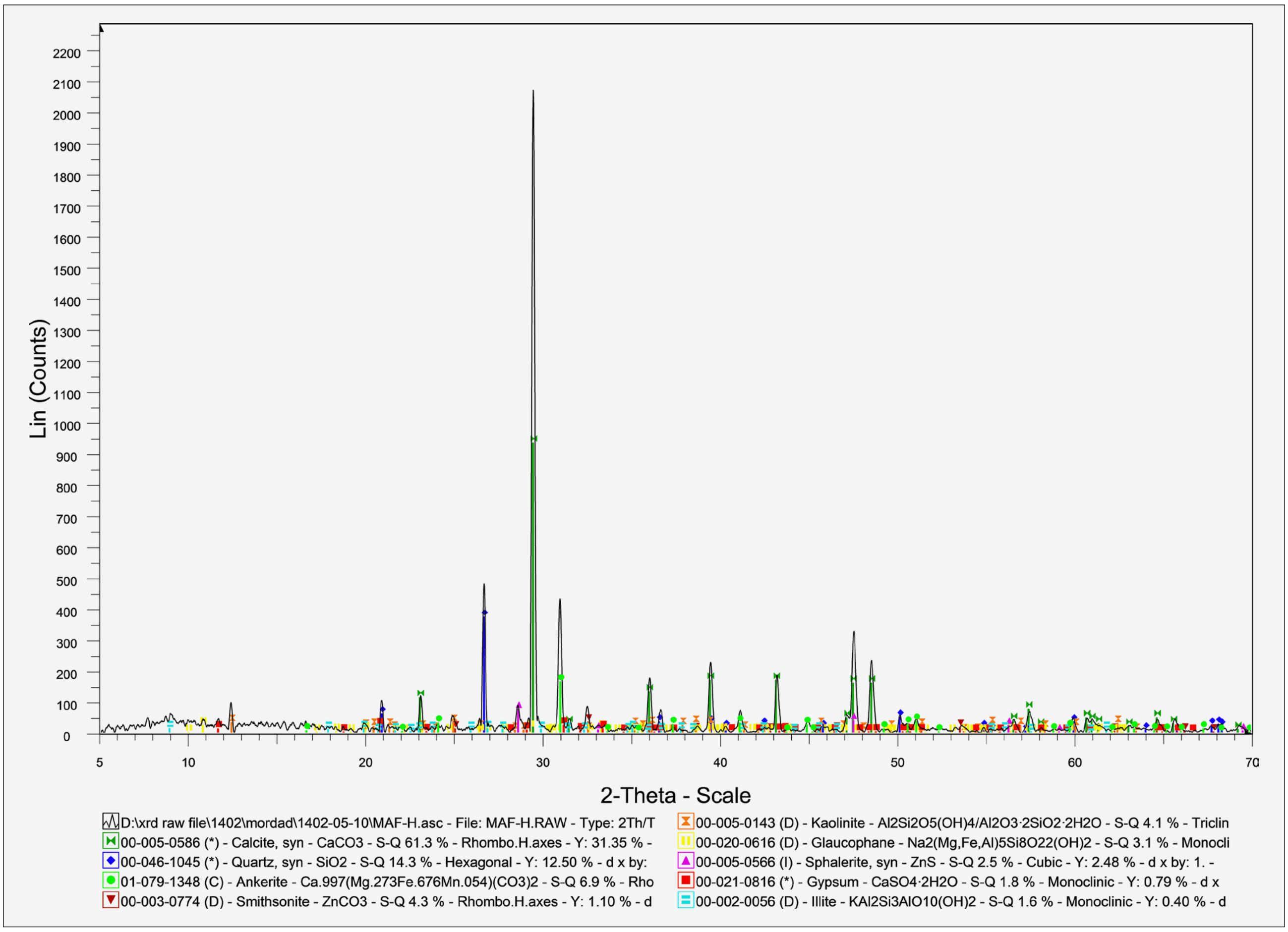

Figure 1. XRD analysis graph

Table 4. Results of ICP-OES analysis

Sample	Ag	Al	As	B	Ba	Bi	Ca	Cd
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	Ppm
	<2	19282	43.6	<10	232	<5	262961	1.4
	Cu	Fe	Ga	K	La	Li	Mg	Mn
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	Ppm
	267	24574	4.3	4016	12.2	12.2	15721	342
	P	Pb	Sb	Sn	Sr	Ti	Tl	V
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	Ppm
	21.95	777.4	<5	<5	482	1018	<5	30.7
	Ce	Co	Cr	Mo	Na	Ni	Zn	S
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
	23	3.6	88.8	<5	4117	27.4	32263	9826

albeit its effect should be investigated in the experiments. In addition, this sample does not contain copper and silver elements.

2.2.1 XRF analysis

In order to identify the elements present in the prepared samples, a representative sample was forwarded for XRF analysis, and its results are reported in **Table 2**.

2.2.2. XRD analysis

In order to identify the minerals present in the prepared sample, the XRD analysis was performed and its results are presented in the form of a graph in **Figure 1** and in **Table 3**.

2.2.3. ICP-OES analysis

To identify the elements present in the prepared sample, the ICP-OES analysis was carried out and its results are shown in **Table 4**.

As can be seen in the XRD, XRF, and ICP analysis tables, the zinc grade is quite clear and zinc minerals including smithsonite and sphalerite have been reported.

2.3. Microscopic studies

In order to prepare polished thin sections, 1 kg of the sample was sieved and samples of different size fractions were sent for the preparation of polished sections. The results of microscopic studies for the size fraction of -106+75 µm are as follows.

In this size fraction, zinc oxide and sulfide minerals (sphalerite and smithsonite) comprise a total of about 2-3% by volume of the fragments, and are often observed in the form of free and separate fragments. Rarely, fragments involving sphalerite and smithsonite can be found. The degree of liberation of zinc minerals in this size fraction (-106+75 µm) was determined to be about 90-95% (see **Figure 2**).

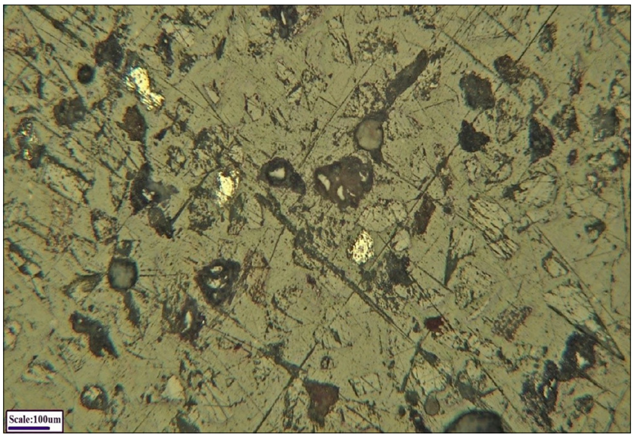

Figure 2. Free fragments of sphalerite, reflected light, PPL condition

Pyrite is present in the range of 0.5-1% by volume, iron oxide-hydroxides in the range of 1-2% by volume, and rarely galena and iron-titanium oxides are mentioned.

2.4. Optimal grinding time

In order to calculate the grinding time, 5 1-kilogram samples were prepared and each was ground by a wet rod mill for specific times of 8, 13, 18, 23, and 28 minutes and with a solids percentage of 50% (one kilogram of sample plus one liter of water). After filtration of the ground samples, 250 g were taken for wet sieve analysis for each time.

Figure 3 presents the graphs of the grinding times versus the crushed d80s. The optimal value of d80 after 8 minutes of grinding was determined. With a grinding time of 8 minutes, the sieve size through which 80% of the material passes was 98 µm.

The bond index of the sample was experimentally estimated, using standard Bond Ball mill and the obtained value was 8.55 kWh/st and/or 9.40 kWh/t, which defines the sample as soft to medium (based on **Table 5**).

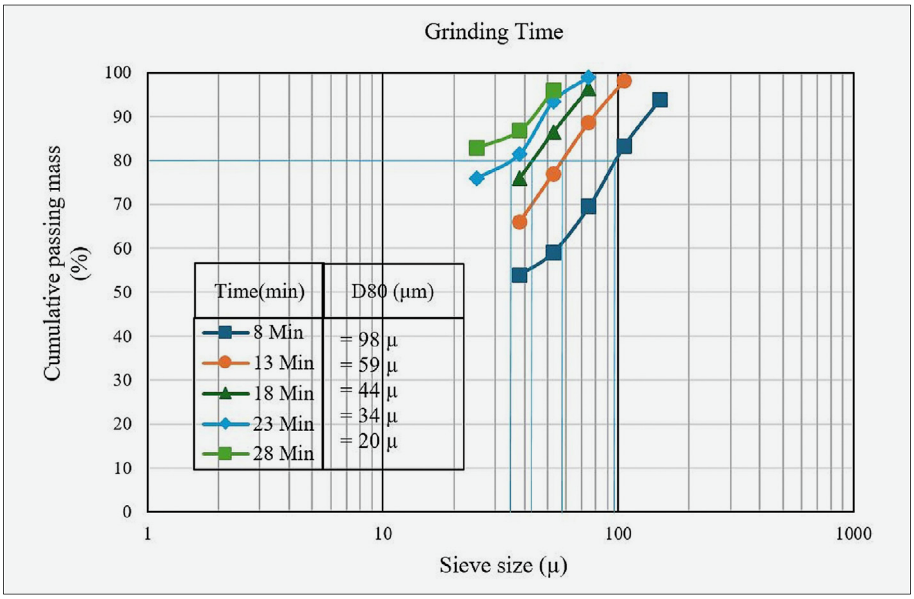

Figure 3. Changes in d80 of crushing curves at different times

Table 5. Classification of rock properties based on bond index value

Property	Soft	Medium	Hard	Very hard
Band index (kWh/st)	7-9	9-14	14-20	>20

2.5. *Flotation tests*

The flotation process is a dynamic process which is established based on the feed and tailings physico-chemical characteristics (**Kazemi et al., 2023),** and operating parameters such as collector concentration, frother concentration, pH, retention time, etc., and the grade of product and recovery could be dramatically changed. Therefore, it is essential to run experiments under different conditions, in order to achieve the optimum values of various parameters.

2.5.1. Initial tests

According to the results of microscopic studies and the desired degree of liberation, which is 80% at a size of 100 µm, 10 flotation tests were defined to obtain the amount of chemical consumption, and the behavior of the feed in the presence of different chemicals. The conditions of these tests are as follows.

In the 1st **experiment**, one kilogram of the sample along with one liter of water was ground in a rod mill for 8 minutes to reach the desired size of particles and degree of freedom. In all ten of these experiments, two skimming stages are performed, the first skimming stage is designed for lead flotation, and the second one is for zinc flotation. In the second stage, a certain amount of chemicals was added to the sample. In this experiment, a 2 liter Denver flotation cell (model D12) with a stirring speed of 1200 rpm and also ethyl xanthate and amyl xanthate collector along with an IRO A65 frother were employed. The conditions of first experiment are given in **Table 6**.

Table 6. First experiment conditions

Conditions	Amount
First stage	
pH of the feed	7.75
ethyl xanthate collector (Z4) with 1% concentration	50 g/t
retention time of the collector	3 minutes
A65 frother	100 g/t
retention time of the frother	1 minute
first skimming stage	5 minutes
Second stage	
amyl xanthate collector (Z6) with 5% concentration	200 g/t
retention time of the collector	3 minutes
A65 frother	60 g/t
retention time of the frother	1 minute
second skimming stage	5 minutes

The 2nd **experiment** was performed similar to the first experiment, but in the first stage, 500 g/t of zinc sulfate was added to depress zinc, and 300 g/t of copper sulfate was added to activate zinc. After each step of addition, the pH was measured, as well. The 3rd **experiment** was in accordance with the conditions of the first experiment, with the difference that sulfidation was also carried out using sodium sulfide with a rate of 1000 g/t. The 4th **experiment** was the same as the second one, but sulfidation was also carried out, using sodium sulfide. In addition, the 5th **experiment** was carried out by eliminating the lead flotation process due to its low grade. The 6th **experiment** was designed from the beginning by eliminating the lead process, due to its low grade and adding the

Table 7. 9th experiment conditions

Conditions	Amount
First stage	
pH of the feed	7.99
Copper sulfate (5% concentration)	250 g/t
retention time of the activator	5 minutes
pH after adding copper sulfate	7.8
amyl xanthate collector (Z6) with 5% concentration	200 g/t
Armax C collector (5% concentration)	250 g/t
retention time of the collector	3 minutes
first skimming stage	10 minutes
Second stage	
copper sulfate (5% concentration)	500 g/t
retention time of the activator	5 minutes
pH after adding copper sulfate	7.8
Armax C collector (5% concentration)	250 g/t
retention time of the collector	3 minutes
second skimming stage	10 minutes

sulfidation step. As the best results were achieved in 5th experiment, so the **7th experiment** was considered identical to the fifth experiment, and after 18 minutes of grinding the feed reached to 40-50 µm, which was finer than other experiments. In the **8th experiment** in accordance with the 5th experiment and with the same amount of grinding time (18 minutes) and by examining the Armax C collector in the second stage, and due to the frothing property of this type of collector, the frother in the second stage was not used. In the **9th experiment**, all the conditions of the previous experiment were maintained and the only change was the addition of the Armax C collector in the first stage. The condition of this test is given in **Table 7**. Finally in the **10th experiment**, it was attempted to use the collector aid which was supplied from South Africa D22-B3 (5% concentration) as a collector aid, for zinc oxide minerals. However, experiment 9 was selected as the experiment with the optimal conditions, in which the zinc grade and recovery were 5 and 70.4%, respectively.

2.6. Flotation kinetics experiment

The flotation kinetic test was conducted to obtain the flotation retention time for maximum recovery in the rougher stage. This test was conducted with the conditions of the 9th flotation test as the optimal test by using 4 kg of the sample with a grinding time of 8 minutes, in 6 skimming stages with specific times. In the first part, after adding chemicals, 4 skimming stages were performed in two periods of 1 minute, one period of 2 minutes and one period of 4 minutes. In the second part, after adding chemicals in a specific amount, 2 skimming stages were performed in periods of 8 minutes and 16 minutes. The results are presented in **Table 8**.

Table 8. Kinetic Test Conditions (4 kg of sample)

Conditions	Amount
First stage	
rotor speed	1200 rpm
pH of the feed	7.99
Copper sulfate	250 g/t
retention time of the activator	5 minutes
amyl xanthate collector (Z6) with 5% concentration	200 g/t
Armax C collector (5% concentration)	250 g/t
retention time of the collectors	3 minutes
Oil	1.2 g/t
Opening of the aeration valve	50%
first skimming stage (0-1)	1 minute
second skimming stage (1-2)	1 minute
third skimming stage (2-4)	2 minute
fourth skimming stage (4-8)	4 minute
Second stage	
amyl xanthate collector (Z6) with 5% concentration	100 g/t
Armax C collector (5% concentration)	125 g/t
Oil	1.2 g/t
The residence time of the above items	1 minute
fifth skimming stage (8-16)	8 minute
Sixth skimming stage (16-32)	16 minute

As the cumulative recovery of the kinetic test was lower than the cumulative recovery of test 9, the kinetic test was repeated for one kilogram of the sample. The conditions are given in **Table 9**.

The results of this test were close to test 9, in terms of cumulative recovery. Therefore, the design of the rougher-cleaner circuit was carried out in accordance with this test, which is accordingly discussed below.

2.6.1. Rougher-Cleaner test

According to the second kinetic test, which was carried out in a one-kilogram sample, 4 kilograms of the sample were prepared. After grinding, each sample was prepared in a one-liter cell so that all tests were similar to the kinetic test. In this test, taking into account the best grade and recovery obtained in the kinetic test, 4 minutes of skimming was carried out, its product was separated as a rougher concentrate, and then, the second stage concentrate was considered as feed of the scavenger stage. The conditions and relevant results are given in **Table 10**.

2.6.2. Closed-cycle Rougher-Scavenger-Cleaner test

The results obtained from the Rougher-Cleaner test were the same with kinetic test in terms of final recovery.

Table 9. Conditions of the second kinetic test (1 kg of sample)

Conditions	Amount
First stage	
rotor speed	1200 rpm
Copper sulfate	250 g/t
retention time of the activator	5 minutes
amyl xanthate collector (Z6) with 5% concentration	200 g/t
retention time of the amyl xanthate collector	3 minute
Armax C collector (5% concentration)	250 g/t
retention time of the collectors	3 minute
Oil	1.2 g/t
first skimming stage (0-1)	1 minute
second skimming stage (1-2)	1 minute
third skimming stage (2-4)	2 minute
fourth skimming stage (4-8)	4 minute
Second stage	
Copper sulfate	500 g/t
retention time of the activator	5 minute
Armax C collector (5% concentration)	250 g/t
retention time of the collectors	3 minute
Oil	1.2 g/t
fifth skimming stage (8-20)	12 minute

Table 10. Rougher-Cleaner test conditions

Conditions	Amount
First stage	
rotor speed	1200 rpm
Copper sulfate	250 g/t
retention time of the activator	5 minutes
amyl xanthate collector (Z6) with 5% concentration	200 g/t
retention time of the amyl xanthate collector	3 minutes
Armax C collector (5% concentration)	250 g/t
retention time of the collectors	3 minutes
Oil	1.2 g/t
first skimming stage	4 minutes
Second stage	
second skimming stage	4 minutes
Copper sulfate	500 g/t
retention time of the activator	5 minutes
Armax C collector (5% concentration)	250 g/t
retention time of the collectors	3 minutes
Oil	1.2 g/t
Continuation of the second skimming stage	12 minutes

Thus, in order to achieve the highest grade and recovery that can represent the plant circuit, the Rougher-Scavenger-Cleaner test was designed in a closed circuit in accordance with the previous test. The discrepancy was that in the first stage, exactly in accordance with the Rougher-Cleaner test, and in the subsequent stages, in addition to the dry feed added in an amount of 3 kg to the Rougher cell, scavenger concentrate and the first cleaner tailings were also added to the 8-liter Rougher cell, as well. The consumed chemicals were used in a ratio of 75% of the initial chemicals. In addition, in the first cleaner in the subsequent stages, in addition to the Rougher concentrate, the second cleaner tailings were also added to it. **Figure 4** presents the block diagram of the Rougher-Scavenger-Cleaner closed circuit.

3. Results and Discussions

Since the main objective of this research was to investigate on the zinc concentration, a number of initial ex-

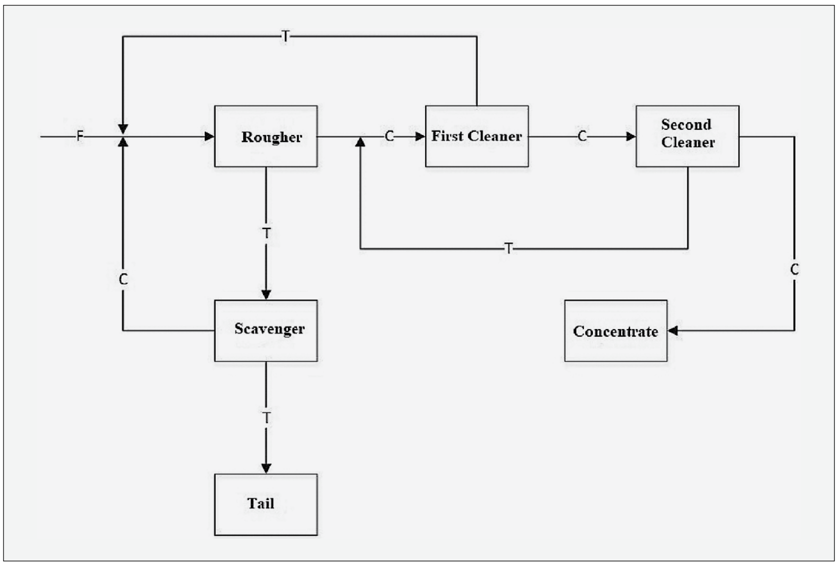

Figure 4. Block diagram of the closed circuit of the Rafter-Scavenger-Cleaner

periments were designed to achieve the best grade and recovery, and to find the optimal flotation conditions. Consequently, after finding the best results, kinetic experiments, rougher-cleaner experiments, and closed-cycle experiments were conducted. The conditions and descriptions of the experiments are presented in the previous section.

3.1. Results of Initial Experiments

In order to find the optimal grade and recovery, 10 initial flotation experiments with changing conditions were performed. The results of these experiments are presented as following.

1st Experiment: After the experiment, the obtained samples including the first and second stage concentrate and tailings were forwarded to the chemical analysis laboratory. The first experiment was designed to determine the behavior of the sample by adding a collector and a frother. Ethyl xanthate collector (Z4) was used for lead flotation and amyl xanthate collector (Z6) was used for zinc flotation. In this experiment, the lead and zinc grades were measured as 0.18 and 6.27%, respectively, and the lead and zinc recoveries were measured as 46.7 and 29.6%, respectively. The results indicated that the test performance in the zinc extraction section was not convienient, and in the lead section, due to the low feed grade, no significant product was obtained, as well.

2nd Experiment: With the conditions of the 1st experiment and adding zinc sulfate in the first stage and copper sulfate in the second stage, the 2nd experiment was designed. In the 2nd experiment, the lead and zinc grades were calculated as 0.29 and 18.32%, respectively, and their recoveries were also calculated as 31.8 and 41.9%, respectively. The results showed that by using zinc sulfate depressant and copper sulfate activator, the grades of both lead and zinc metals increased and also by activating the surface, the recovery of zinc increased, but the recovery of lead was still not significant due to its low grade in the feed.

3rd Experiment: In this experiment, by maintaining the conditions of the 1st experiment and adding sodium sulfide in both stages, the sulfidation process was applied to sulfide the surface of oxide minerals. In general, the process of combining or saturating with sulfur or its compounds is called sulfidization. Initially, only the sulfite hydrate ion was used as an activator of oxide minerals until Schwartz published the results of his studies on the sulfidization of the surface of oxide minerals in 1905 (**Moradi, 2005**). In this experiment, the lead and zinc grades were calculated to be 0.27 and 2.78%, respectively, and the lead and zinc recoveries were calculated to be 30.6 and 6.8%, respectively. According to the results, the sulfidation process for the lead increased the concentrate grade and decreased the recovery compared to the first experiment, which cannot be justified, but it had a negative effect for the zinc, so that the grade and recovery dropped sharply, which could indicate the depression of zinc that was not flotated and activation did not take place in this section.

4th Experiment: By combining the conditions of the 2nd and 3rd experiments, namely the use of sodium sulfide, zinc sulfate and copper sulfate, the 4th experiment was conducted in two stages. In this experiment, the lead and zinc grades were calculated as 0.42 and 3.03%, respectively, and the lead and zinc recoveries were calculated as 37.9 and 5.1%, respectively. The results showed that the lead grade and recovery were better than the previous experiments, and it would therefore be postulated that the zinc retention and sulfidation process in the lead section had better results, but it is still not justified due to the low lead product grade. Of course, the zinc activation and sulfidation process in the second section was not desirable, and it can be concluded from the conditions of the 2nd and 4th experiments that the depression and activation process in the zinc section was not effective. The sulfidation operation also had a negative effect on this section, and the zinc grade and recovery dropped sharply.

5th Experiment: As the results of the experiments show, the lead grade was very low and the enrichment of this metal for the project will not be economically viable. Therefore, the analysis of lead metal has been omitted in the following and the experiments have been focused on the zinc. According to the comparison of the 1st and 2nd experiments to investigate on the zinc activation operation, and its positive effect on zinc concentration, the first stage of this experiment was carried out by using copper sulfate as a zinc activator in the amount of 250 g/t and using twice the amount of A65 frother compared to the 1st experiment in the amount of 200 g/t, as well as the amyl xanthate collector in the amount of 200 g/t. In the second stage, in addition to the mentioned chemicals, the sulfidation process was also applied. The amount of copper sulfate was twice the amount in the first stage and the frother was used in the amount of 140 g/t. By applying these conditions, the grade and recovery of zinc were obtained as 12.82 and 40.2%, respectively. The results show that increasing the skimming time to 10 minutes, and using the above chemicals increased the zinc content by almost two times compared to the 1st experiment. Zinc recovery also increased significantly.

6th Experiment: In this experiment, the conditions of 5th experiment were constant and in addition to the second stage, the sulfidation process was also carried out in the first stage. In this experiment, the zinc grade and recovery were 4.7 and 18.5%, respectively, which were significantly lower than the zinc grade and recovery in the previous experiment. Therefore, according to the results of several experiments with different conditions, the use of sodium sulfide as a sulfidation agent for this sample is not recommended, as it decreased zinc grade and recovery.

Table 11. Zinc results in the 9th experiment

Sample	Mass (gr)	Mass (%)	Grade and Recovery (%)				
			Oxide		Total		Oxide to Total Ratio (%)
			grade	Recovery	grade	Recovery	
First stage concentrate	280.2	28.3	1.48	22.6	6.52	55.1	22.7
Second stage concentrate	185.3	18.7	2.41	24.3	2.72	15.2	88.6
Tailing	525	53	1.86	53.1	1.87	29.6	99.5
Feed	990.5	100	1.86	100	3.34	100	55.5

Table 12. Results of the kinetic test

Steps	Sample	Skimming time (min)	Mass (gr)	Mass (%)	Total grade (%)	Recovery (%)	Cumulative mass (%)	Average grade (%)	Cumulative recovery (%)	Enrichment Ratio
First stage	First concentrate	1	96.1	2.4	3.95	3.12	2.4	3.95	3.12	1.28
	Second concentrate	1	109.4	2.8	4.36	3.92	5.2	4.17	7.05	1.35
	Third concentrate	2	189.7	4.8	7.59	11.85	10	5.81	18.9	1.89
	Fourth concentrate	4	205	5.2	5.82	9.82	15.2	5.81	28.71	1.89
Second stage	Fifth concentrate	8	84.8	2.1	6.55	4.57	17.4	5.91	33.28	1.92
	Sixth concentrate	16	534	13.5	3.79	16.65	30.9	4.98	49.94	1.62
-	Total tailing	-	2728.5	69.1	2.23	50.06	100	-	-	-
-	Feed	-	3947.5	100	3.08	-	-	-	-	-

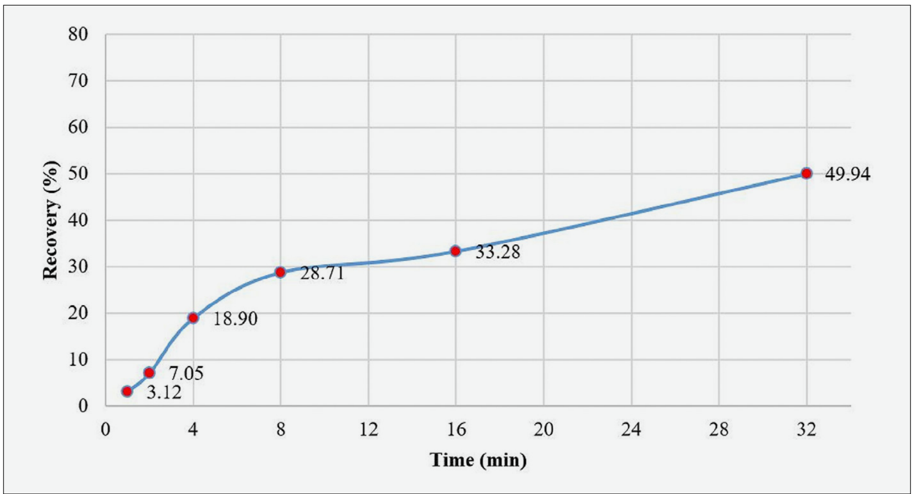

Figure 5. Recovery variation curve of the first kinetic test versus the grinding time

7th Experiment: According to the results of the 5th experiment as an experiment with desirable grade and recovery, the 7th experiment was designed with the conditions of the 5th experiment. In order to investigate the effect of particle size on grade and zinc recovery, the materials were re-ground with a grinding time of 18 minutes until they reached to size of 40-50 µm. The grade and zinc recovery of this test, were calculated as 7 and 52.3%, respectively. The results show that reducing the particle size causes a decrease in grade and an acceptable increase in zinc recovery; this result could stem from the phenomenon of zinc *entrainment* in fines sizes that has caused this issue.

8th Experiment: By removing sodium sulfide from the conditions of 5th experiment, and adding the Armac-C collector in the second stage, 8th experiment was conducted. It should be noted that due to the frothing property of the Armac-C collector, the addition of the A65 frother in the second stage was avoided. With these conditions, the zinc grade and recovery were 3.9 and 66.1%,

Table 13. Results of the second kinetic test (one kilogram)

Steps	Sample	Skimming time (min)	Mass (gr)	Mass (%)	Total grade (%)	Recovery (%)	Cumulative mass (%)	Average grade (%)	Cumulative recovery (%)	Enrichment Ratio
First stage	First concentrate	1	24.2	2.4	34.36	26.66	2.4	34.36	26.66	11.16
	Second concentrate	1	21.4	2.2	10.24	7.03	4.6	23.04	33.68	7.48
	Third concentrate	2	37	3.7	4.72	5.6	8.4	14.83	39.28	4.82
	Fourth concentrate	4	53.8	5.4	2.85	4.92	13.8	10.11	44.2	3.28
Second stage	Fifth concentrate	12	187.4	18.9	2.4	14.42	32.7	5.65	58.62	1.83
-	Total tailing	-	665.3	67.3	1.94	41.38	100	-	-	-
-	Feed	-	989.1	100	3.15	100	-	-	-	-

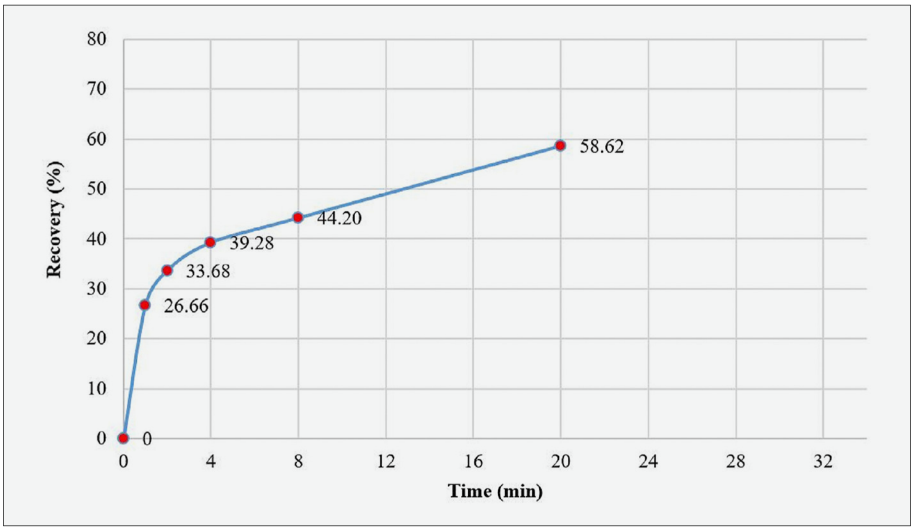

Figure 6. Recovery variation curve of the second kinetic test versus grinding time

respectively, which significantly reduced the grade and increased recovery compared to the results of 5th experiment. This experiment had a higher recovery compared to 7th experiment which had a reduction in grain size, and the purpose of the rougher experiments was to increase recovery. As mentioned above, the reason for the reduction in grade was the zinc *entrainment*, but in this experiment, the reason for the reduction in grade was the increase in recovery in the second section, which could be considered the effect of the Armac-C collector in collecting as much zinc as possible in the second stage.

9th Experiment: In this experiment, by eliminating the sulfidation agent due to its negative effect and using chemicals that had positive results in previous experiments, such as Armac-C collector and copper sulfate, it was attempted to achieve the highest zinc grade and recovery. The results of the 9th experiment are shown in **Table 11**.

According to the calculations, the zinc grade and recovery in this experiment were calculated as 5 and 70.4%, respectively. Due to the proper performance of the Armac-C collector, this collector was also used in the first section, and the frother was removed due to the frothing property of the collector in both stages. This experiment was determined as an experiment with optimal conditions with the methods and chemicals mentioned.

10th Experiment: In this experiment, along with the amyl xanthate collector, the D22-B3 collector aid was supplied and used. The zinc grade and recovery in this experiment were 13.94 and 37.1%, respectively. Compared to the previous experiment, the zinc grade increased, but the recovery did not decrease. Therefore, the collector aid used in this experiment was not suitable. The purpose of designing and conducting the initial experiments was to achieve the highest recovery percentage and with a view to the grade. Since the 9th experiment has a higher recovery than the other experiments, the conditions of this experiment are selected as the optimal experiment.

3.2. Results of the kinetic test

According to the conditions of the 9th experiment as the test with optimal conditions, the kinetic test was car-

Table 14. Results of the Rougher-Cleaner Test

Sample (duration minutes)	Mass (g)	Mass (%)	Cumulative mass (%)	Total Zinc		Oxide Zinc		Ratio
				Grade (%)	Recovery (%)	Grade (%)	Recovery (%)	
Concentrate of Cleaner II (0-1)	32.8	0.8	0.8	52.81	14.5	0.17	0.1	0.3
Concentrate of Cleaner II (1-2)	5.8	0.1	1	41.8	2	0.37	0.03	0.9
Concentrate of Cleaner II (2-3)	3.7	0.1	1.1	26.13	0.8	0.71	0.04	2.7
Concentrate of Cleaner II (3-4)	3.1	0.1	1.2	17.34	0.4	0.82	0.04	4.7
Tailing of Cleaner II (0-4)	46.2	1.2	2.3	4.3	1.7	1.48	1	34.4
Tailing of Cleaner I (0-4)	244.5	6.2	8.5	3.59	7.3	1.48	5.1	41.2
Scavenger Concentrate (4-20)	1100	27.9	36.4	3.35	30.8	1.78	27.8	53.1
Scavenger Tailing (4-20)	2510	63.6	100	2.02	42.4	1.85	65.9	91.6
Computational feed	3946.1	100	-	3.03	100	1.79	100	58.9

Table 15. Results of the Rougher-Scavenger-Cleaner test

Sample	Mass (g)	Mass (%)	Cumulative mass (%)	Total Zinc		Oxide Zinc		Ratio
				Grade (%)	Recovery (%)	Grade (%)	Recovery (%)	
Concentrate of Cleaner II	252	1.7	1.7	42.19	23.05	0.4	0.53	0.95
Tailing of Cleaner II	90	0.6	2.3	9.56	1.87	1.15	0.54	12.03
Tailing of Cleaner I	1940	13	15.3	2.78	11.69	1.56	15.82	56.12
Scavenger Concentrate	2769	18.5	33.8	3.75	22.51	1.53	22.14	40.8
Scavenger Tailing	9912	66.2	100	1.9	40.89	1.18	60.97	61.83
Feed	14963	100	-	3.08	100	1.28	100	41.47

Table 16. Final Grade and Recovery Results

Feed grade (%)	Concentrate grade (%)	Tailing grade (%)	Total recovery (%)
3.08	42.19	1.9	40.08

ried out, using 4 kg of the sample, and the relevant results are shown in **Table 12**, and in **Figure 5**.

Given that the cumulative recovery of the kinetic test was calculated to be less than the cumulative recovery of the 9th experiment, which was considered the optimal test, the kinetic test was repeated for one kilogram of the sample. The results of the second kinetic test are as described in **Table 13**, and **Figure 6**.

The second kinetic test had a higher recovery than the first test and reached a recovery closer to the 9th experiment. The purpose of the kinetic test was to achieve the retention time in the rougher section, which should have the highest recovery in this section. Therefore, according to the above results, an approximate recovery of 59% could be achieved in this kinetic test.

3.3. Results of the Rougher-Cleaner Test

The results of the Rougher-Cleaner test with the conditions mentioned in section (2.6.1) are shown in **Table 14**.

After achieving the maximum recovery according to the retention time of the kinetic test, the cleaner operation was also carried out on the rougher stage concentrate, and in each stage, the concentration was carried out for 1 minute, so that the kinetics of the cleaner stage could reach the maximum recovery, and the appropriate grade in this section, accordingly. The results show that after four stages of concentration for 4 minutes, a grade of 46.8% was obtained. Considering the grade of the first cleaner tailings and the scavenger concentrate, which was close to the grade of the input feed, a closed-circuit test was designed to calculate the grade and final recovery, which can be representative of industrial conditions, during several closed-circuit flotation stages.

3.4. Results of the Rougher-Scavenger-Cleaner test

The results of the Rougher-Scavenger-Cleaner test with the conditions mentioned in section (2.6.2) are shown in **Table 15**.

The results in **Table 15** were obtained with 15 kg of feed prepared according to the optimal conditions, and after 5 stages of closed-circuit flotation test to achieve a steady state, which shows that the zinc concentrate grade was desirable, but the zinc grade in the tailings was high, which was also the reason for the reduced recovery. Therefore, according to the designed test, the flotation process was completed, and in order to reduce the zinc grade in the tailings, which was mostly in the oxide phase, a leaching test should be designed. Thus, the final grade and recovery of the flotation section are as follows in **Table 16**.

As can be seen in **Table 16**, the feed grade and concentrate of zinc is 3% and 42.19%, respectively, and the overall recovery is 40.08%.

4. Conclusions

1) The results of XRD and XRF analyses showed that the lead and zinc ore sample from the Nignan deposit has a zinc grade of 3.01% and lead between 0.04 and 0.05%, and carbonate minerals (calcite, less dolomite and iron carbonates) are the main non-metallic phase.
2) The results of microscopic and degree of liberation studies showed that this sample contains zinc sulfide and oxide minerals, including sphalerite and smithsonite, and has 90 to 95% of liberation in size fraction of 90-100 μm.
3) The results of the grinding time tests indicated that the sample reached a degree of liberation of about 98 μm in 8 minutes of grinding by a wet rod mill.
6) According to the sieve analysis of the feed and product of the Bond ball mill, the Bond work index was obtained as 9.40 kWh/t (8.55 kWh/st), which defines that the sample was in the soft to medium category.
7) Initial flotation tests were designed in 10 experiments, and the best result in terms of grade and recovery was considered from the 9th experiment, in which 250 g/t of copper sulfate, 200 g/t of 5% amyl xanthate collector and 250 g/t of Armac-C collector aid were employed in the first stage. In the second stage of this test, 500 g/t of copper sulfate and 250 g/t of Armac-C collector were used.
8) The zinc grade and recovery in the 9th experiment were obtained as the best experiment, 5 and 70.4%, respectively.
9) Due to the low lead content in the feed, the lead section was omitted from the flotation tests.
10) Kinetic tests were designed and run to calculate the highest recovery in the rougher section.
11) According to the results of the kinetic tests and the rougher-cleaner test, the closed-circuit flotation test was designed which represents the industrial process.
12) The final grade and recovery of flotation were obtained as 42.19 and 40.08% for zinc concentrate, respectively.

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SAŽETAK

Koncentriranje uzorka olova i cinka iz rudnoga ležišta Nignan

Cilj je ovoga istraživanja flotacijom povećati udio cinka na više od 40 % iz olovne i cinkove rude iz područja Noahang (Chah-Ahan) ležišta Nignan. Eksperimenti su provedeni na uzorku s prosječnim udjelom od 3 % cinka i 0,05 % olova. Glavni minerali cinka u ovome uzorku bili su karbonati sfalerit i smitsonit, a galenit je rijetko uočen. Rezultati mineraloških istraživanja na poliranim tankim izbruscima pokazali su da je mogući stupanj raščina cinkovih ruda 90 – 95 % u frakciji veličine –106+75 μm. Istraženi su različiti parametri poput veličine, postotka čvrste mase, vrste i doziranja kolektora te je pod optimalnim uvjetima dobiven koncentrat s udjelom od 5 % cinka i iskorištenjem od 70,4 %. Također, provedeni su kinetički eksperimenti kako bi se postiglo najveće iskorištenje cinka u grubljoj fazi, koje je dosegnulo 58,6 %. Rezultati su pokazali da su najbolji stupanj i iskorištenje cinka postignuti kada je uzorak drobljen mokrim štapnim mlinom 8 minuta kako bi se postigao željeni stupanj raščina. Flotacijom je postignuta konačna kvaliteta koncentrata od 42,19 % i iskorištenje od 40,08 % za ispitivani koncentrat cinka.

Ključne riječi:
mineralno područje Noahang, flotacija, stupanj raščina, kvaliteta koncentrata, iskorištenje

Author's contribution

Maryam Abedi Moghadam (Master's degree) gathered samples, characterized the samples, and contributed to the crushing, grinding, separation and flotation processes and described the characterization and evaluation of the final products. **Marzieh Hosseini Nasab** (Associate Professor) also contributed to the crushing, grinding, separation and flotation processes. Supervision, validation, visualization, writing – original draft and writing – review & editing were her other responsibilities for this research. **Mohammad Noaparast** (Professor of Mineral Processing) evaluated the results, reviewed the draft manuscript, and provided technical suggestions.
All authors have read and agreed to the published version of the manuscript.