

Beneficiation of Artvin-Cerattepe copper-zinc ore by flotation

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Abstract: The Cerattepe (Artvin) mine contains volcanogenic massive sulfide (VMS) deposits in northeastern Turkey. This is a kuroko type deposit located in Late Cretaceous volcanic, intrusive and sedimentary rocks, and has a structure with dense alteration stages. In this study, batch flotation tests were carried out to determine the flotation behavior of a new sample (mix ore), that was the combination of two different ores with different flotation behaviors. The mixed ore contains 2.18% Cu, 1.46% Zn, (Cu/Zn ratio 1.49) 38.35% Fe, and 41.6% S content. An efficient separation of copper minerals from zinc minerals using the conventional selective flotation method could not be achieved due to complex mineralogy and very low liberation degree. Therefore, sequential selective flotation resulted in poor Cu recovery. The effects of flotation parameters such as collector type, collector amount, particle size, and pH conditions, were investigated to obtain a bulk copper-zinc concentrate. The best results were obtained using thionocarbamate (Aero 3894) and dithiophosphine (Aerophine 3418A) collector reagents at $d_{80}=40\ \mu\text{m}$ particle size. Under optimal conditions (grain size, $d_{80}=40\ \mu\text{m}$, pH=11, amount of collector reagent 60 g/t (Aero 3894), frother (MIBC)=50 g/t, solid ratio=32%, flotation time=8 min.), a bulk copper-zinc concentrate containing 13% Cu and 9.5% Zn was obtained with a copper yield of 84.4% and a zinc yield of 88.9%. The concentrate mass pull was 13%. After rougher and two-stage scavenger flotation, a concentrate (rougher concentrate + scavenger products) was obtained with a mass pull of 22%. Copper and zinc recoveries of this concentrate were 91.8% and 92.5%, respectively.

Keywords: copper, zinc, flotation, collector, refractory ore

1. Introduction

Metals were discovered and first used approximately 10,000 years ago. Copper was the first metal to be used as a substitute for stone by humans and has remained one of the most important materials in the development of civilization. Copper has become a major industrial metal, ranking third after iron and aluminum in terms of quantities consumed due to its properties, usage, both singularly and in combination, its high ductility, malleability, and thermal and electrical conductivity, and its resistance to corrosion and antimicrobial activity. Electrical uses of copper, such as power transmission and generation, building wiring, telecommunication, and electrical and electronic products, account for about three quarters of total copper use today. Building construction is one of the largest copper-using markets, followed by electronic products, transportation, industrial machinery, and consumer and general products. In addition, copper is one of 17 critical metals used across a variety of clean energy generation (solar and wind technology) and storage technologies (flow, lithium-ion batteries and sodium batteries), and electric vehicles. The demand for copper is therefore expected to increase continuously to create a low-carbon future. Furthermore, the advent of energy storage technologies, the transition to wind and solar energy, and the increase in electric vehicles promises to increase copper usage in the future. Copper is therefore considered to be one of the critical material components for the next great energy transition (Schipper, 2018; Hund et al., 2020; Park et al., 2021; Aikawa et al., 2022). The depletion of rich-grade copper ores and continuously increasing demand make the processing of low-

grade or complex ore resources both inevitable and mandatory for sustaining industrial growth and development.

Flotation is the most commonly used method for recovering copper minerals from sulfide ores and gangue (Fuerstenau et al., 2000; Khmeleva et al., 2002; Ejtemaei and Nguyen, 2017; Huang et al., 2019; Han et al., 2020). However, refractory copper-zinc sulfide ores are difficult to treat due to their complex mineralogy and high levels of gangue minerals. These ores typically require higher collector dosages, more selective collectors, careful control of pH, solid ratio, and other process parameters, as well as detailed mineralogical studies to achieve satisfactory flotation performance. The most common copper sulfide flotation collectors are the hydrogen sulfide (-SH) class, also known as thiols, sulfhydryl group, or mercapto group (C-S). These can be grouped into the families of xanthate (X), dithiophosphate (DTP), dithiocarbamates (DTC), thionocarbamate, thiocarbamate, mercapto benzothiazole (MBT) and phosphine-based collectors (Castellón et al., 2022). The different collectors (single or mixed) and their interactions on the recovery and grade for copper sulfide flotation have been studied by several researchers (Ackerman et al., 1987; Bradshaw, 1998; Bagci et al., 2007; Lotter and Bradshaw, 2010; McFadzean et al., 2012; Chen et al., 2014; Hassanzadeh and Hasanzadeh, 2016; Molaei et al., 2018; Dhar et al., 2019a; Dhar et al., 2019b; Bazmandeh and Sam, 2021).

Massive copper-zinc sulfide ore is a complex and refractory polymetallic ore resource; thus, ascertaining its mineralogical properties is crucial for comprehensive recovery of valuable minerals (Xie et al., 2014). Mineral processing is often complex, and recovery can be poor. Many flowsheets are often used in the separation of copper-zinc sulfide ores, such as sequential flotation, bulk flotation, and two-stage flotation (Xie et al., 2014; Bulatovic, 2015).

Process mineralogy has great importance in terms of determining the beneficiation process of a complex or refractory ore (Çelik et al., 2011; Bradshaw, 2014; Nayak et al., 2020; Birinci and Gök, 2021; Altınışık et al., 2022). Recent investigations have shown that effective flotation of such complex ores requires process mineralogy data to be used in developing flotation flowsheets, setting appropriate reagent regimes, and determining liberation degrees (Yang et al., 2016; Bahrami et al., 2019; Zhang et al., 2020; Nghipulile et al., 2022). The steps of this process are determined on the basis of detailed mineralogy studies (Zhao et al., 2022). In particular, technological devices (MLA, electron microscopes, etc.) developed recently have been successfully used in all steps of ore enrichment processes (Can et al., 2018; Chen et al., 2018; Whiteman et al., 2016).

The Cerattepe Deposit is a kuroko-type VMS deposit located in Late Cretaceous age volcanic-sediment and intrusive host rocks (Akpınar and Çiftçi, 2019; Ucurum et al., 2021); it is distinguished by an unusual basal zone of high-grade copper sulfides and an overlying Au-rich oxide zone from the other VMS deposits of northeastern Pontides. The mineral paragenesis of the Cerattepe VMS deposit comprises pyrite, sphalerite, marcasite, chalcopyrite, bornite-idaite, galena, covellite, chalcocite, cubanite, cuprite including sulfosalts (mainly tennantite and lesser tetrahedrite) gold, silver, arsenopyrite, and bournonite. Gangue minerals include barite, quartz, gypsum, anhydrite, and calcite. Hematite, limonite, lepidocrocite, malachite and azurite, and jarosite are the oxidation minerals (Akpınar and Çiftçi, 2019; Çiftçi, 2019). The VMS deposits have been a major source of copper and, to a lesser extent, of zinc and lead, and have made important contributions to the economic development of Turkey for over half a century. Copper-zinc sulfide ore in Cerattepe is a complex and refractory polymetallic ore resource, and primarily contains four copper minerals (chalcopyrite, chalcocite, bornite, and covellite), sphalerite and pyrite. In this study, the mineralogical properties of this ore were characterized in detail following a multidisciplinary approach, including chemical (inductively coupled plasma optical emission spectroscopy, ICP-OES), phase, X-ray diffraction (XRD), scanning electron microscopy (SEM), optical microscopy, and mineral liberation analyses (MLA). MLA and SEM analyses revealed that copper and zinc minerals have a very low liberation degree and the presence of 'chalcopyrite disease' (fine-grained chalcopyrites of micron and submicron sizes in sphalerite). A series of selective flotation experiments were designed using a large variety of collectors, depressants, and high dosages, but the effective separation between copper minerals and sphalerite could not be significantly enhanced even at fine particle size ($d_{80}=12\ \mu\text{m}$). Based on the systematic mineralogical properties obtained, a bulk flotation method was proposed to recover copper-zinc minerals from complex sulfide ore. Therefore, bulk flotation tests were performed in this study to investigate the

influence of residence time, collector type and concentration, particle size, and pH on flotation performance.

2. Materials and methods

2.1. Material

The sulfide copper-zinc ore sample used in this study (approximately 200 kg) was obtained from the Eti Bakır Murgul operation Cerattepe (Artvin) deposit. Elemental analysis of the ores was performed by the wet chemical analysis method using inductively coupled plasma optical emission spectrometry (ICP-OES), the results of which are shown in Table 1. The sample contained 2.18% Cu, 1.46% Zn, 0.33% Pb, 38.35% Fe, 41.60% S, and 0.87 g/t Au.

Raw ore was prepared for the experimental test work by jaw crushing to obtain a representative sample of material sized 10 mm. This sample was then blended and split into four samples of 50 kg. The 50-kg sample was further crushed to obtain a 4-mm sample size using a jaw crusher. These samples were then homogenized, riffled, and split into subsamples of 1 or 2 kg each.

Table 1. Chemical analysis results for the Cerattepe ore sample

Element	Unit	Sample content	Element	Unit	Sample content	Element	Unit	Sample content
Ag	ppm	27.34	K	%	0.06	Sb	ppm	180
Al	%	0.22	Mg	%	0.10	Sc	ppm	0.93
Au	ppm	0.87	Mn	%	0.01	Th	ppm	217
Ba	%	0.14	Mo	ppm	217	Ti	ppm	118
Bi	ppm	90	Na	%	0.03	U	ppm	1297
Ca	%	0.49	Ni	%	0.01	Y	ppm	0.98
Co	%	0.02	P	ppm	69.06	Yb	ppm	2.51
Cu	%	2.18	Pb	%	0.33	Zn	%	1.46
Fe	%	38.35	Rb	ppm	384	SiO ₂	%	3.13
Hg	ppm	44.36	S	%	41.60	LOI	%	29.13

2.2. Methods

Mineralogical characterization was first carried out on the raw ore using optical microscopy and X-ray diffraction (XRD) to identify both composite particles and the various mineral phases in the ore. The XRD patterns were recorded on a Rigaku X-ray diffractometer using CuK α radiation with a scanning rate of 2° min⁻¹ from 3° to 70°. The generator voltage and current were 40 kV and 30 mA, respectively. Rietveld refinement analysis using X'Pert HighScore Plus software (PANanalytical) was performed to obtain the percentages of different phases in the samples. Samples of polished sections were analyzed using a reflected-transmitted polarizing light microscope (Nikon E400POL with Nikon Coolpix 995 digital camera). Polished samples for ore mineralogy were prepared by a Struers TegraPol-15 grinding/polishing machine with variable speeds (40–600 rpm in steps of 10 rpm) and with a disc 200 mm in diameter. The modal mineralogical compositions, calculated assays, elemental distributions, mineral locking, and physical properties of the ore samples were determined by MLA. MLA analysis was performed using FEI MLA 650 and FEI Quanta 400 MLA instruments with a Bruker X-Flash dual EDS system. The MLA Suite 3.1 program package and FEI Standard Reference, FEI Mineral Reference 20 kV, and FEI Reference STD 25 kV mineral libraries were used for the data creation, processing, and display operations. Particle size analysis was performed using a Partica LA-950V2 laser diffraction particle size distribution analyzer (Horiba) in wet mode. Elemental analysis of the solid sample was performed by inductively coupled plasma optical emission spectrometry (ICP-OES) (Varian 710-ES).

Batch flotation experiments were carried out using a Denver flotation machine with a solid percentage of about 32% w/w at the desired impeller speed (900–1300 rpm) using a 2.5 dm³ flotation cell. A freshly ground ore (d₈₀:60, 40 and 20 μ m) was used in all flotation experiments, and all experiments were conducted using tap water. Lime (Ca(OH)₂) or sulfuric acid (H₂SO₄) were used to

modify the target pH value during the flotation process, after which the flotation agents were added to the slurry with a 5 min and 1 min conditioning time for the collector, depressant/activator and frother, respectively. At the end of the flotation experiment, all the concentrate products and tailings were filtered and dried in an oven at 105 °C for 24 h before being subsequently weighed for suitable analysis.

Sequential flotation: A series of tests for selective separation of copper-containing minerals from sphalerite and pyrite was carried out. The ore was wet ground to $d_{80}=20\ \mu\text{m}$ and $d_{80}=12\ \mu\text{m}$ in size. In these tests, the effects of various depressants such as NaCN, Na_2SO_3 , $\text{Na}_2\text{S}_2\text{O}_5$, Na_2S , ZnSO_4 at different pH values were investigated for the depression of sphalerite. Aerophine 3418A and MIBC were used as collector and frother.

Bulk flotation: The effect of different collectors has been examined for rougher/scavenger flotation to obtain high copper recovery. In addition to the three different particle sizes used. The samples were also tested at two different pH environments (9.5, and 11.0). Sodium isopropyl xanthate (SIPX), potassium amyl xanthate (KAX), and potassium ethyl xanthate (KEX) were thiol-type conventional collectors used sulfide flotation. Aerophine 3418A is an aqueous solution of sodium diisobutyl dithiophosphinate and has been widely used in the flotation of copper and lead-sulfide minerals, particularly those associated with complex sulfide ores containing sphalerite mineralization, and ores with high levels of pyrite and/or pyrrhotite (Cytec, 2010; Pecina-Trevino et al., 2003; Bulut et al., 2021). Moreover, collectors such as Aero 3894 (dialkyl thionocarbamate), Aerofloat 238 (alkyl dithiophosphate) (from Cytec Solvay) were also tested. The flowsheet of flotation experiments is shown schematically in Fig. 1.

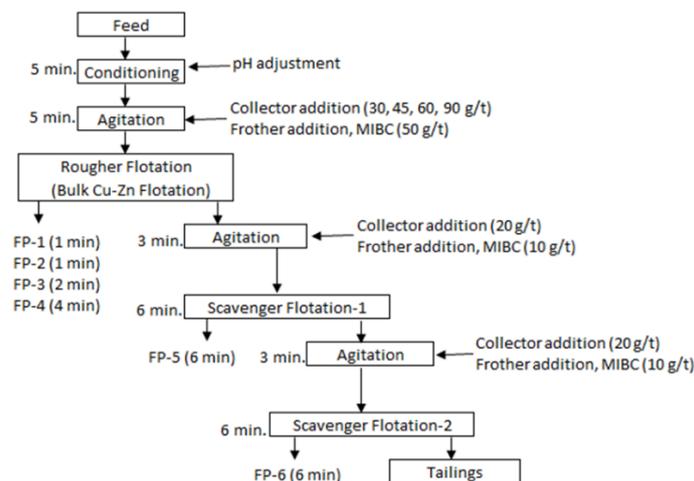


Fig. 1. The flowsheet of bulk flotation experiments

3. Results and discussion

3.1. Mineralogical studies

Microscopic studies are the most important methods for the selection of separation methods (Molaei et al., 2018). These can be applied for liberation degree to confirm the XRD studies and determine some ore properties such as grain zone, shape, texture, and intergrowth of minerals (Molaei et al., 2018). In the representative sample, major minerals such as pyrite (FeS_2), chalcopyrite (CuFeS_2), chalcocite (Cu_2S), sphalerite (ZnS), and barite (BaSO_4) were detected by XRD (Fig. 2). The main mineral composition of the ores and mineral liberation degree in the ores determined by mineral liberation analyzer (MLA) measurements are provided in Table 2 and Table 3, respectively. More than 50 minerals were identified by MLA. Table 2 shows the mineral distribution on a size-by-size basis for the ore sample. The main copper-bearing minerals are chalcopyrite, chalcocite, bornite, and covellite, and lesser amounts of tetrahedrite and copper sulfate, among which barite and quartz are the major oxide gangue minerals.

Pyrite showed higher liberation, with a value of more than 90%. However, copper containing minerals and sphalerite had a very low liberation degree and these minerals are mostly associated with pyrite (Table 3). The percentage of free particles increased to a small extent at $-45 + 20\ \mu\text{m}$ size fraction

in all copper minerals. In the -45+20 μm size fraction, the liberation degrees of chalcopyrite, covellite, chalcocite, bornite and sphalerite, were only 42.0%, 38.3%, 26.5%, 19.3% and 55.8%, respectively. A significant part of sphalerite was in the form of binary association with pyrite, chalcopyrite, and barite. Copper minerals generally showed interlocking with pyrite, then with sphalerite, and then with one another; it is therefore impossible to achieve high-efficiency selective flotation for copper and zinc minerals.

The chalcopyrite inclusions observed within the sphalerite reflect co-precipitation from the same hydrothermal solution. Fine-grained chalcopyrite with micron and submicron sizes, which develop under conditions similar to those of the sphalerite in which they occur (temperature, salinity, sulfur

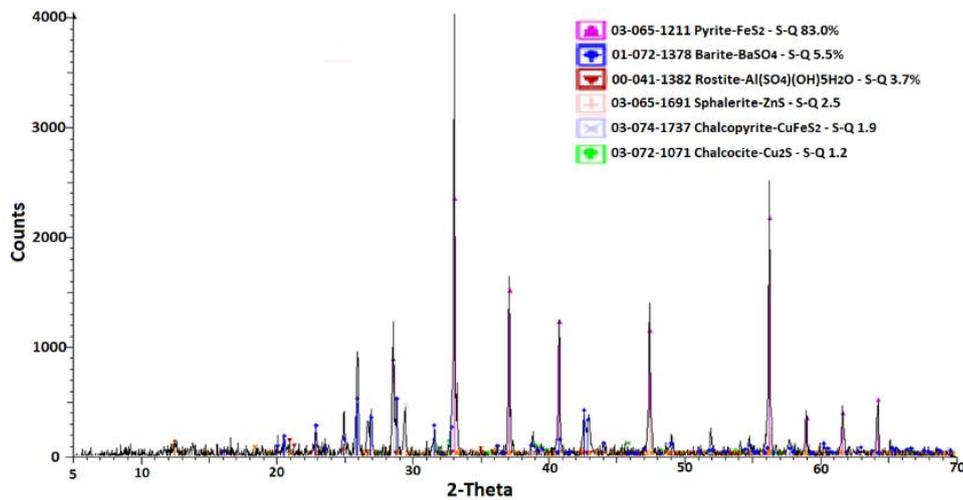


Fig. 2. XRD pattern for Cerattepe copper-zinc ore

Table 2. The modal mineralogy of ore

Mineral	Formula	Wt%	
		-75+45 μm	-45+20 μm
Pyrite	Fe^{2+}S_2	87.72	86.12
Sphalerite (Medium Fe)	$(\text{Zn},\text{Fe})\text{S}$	1.58	1.70
Barite	$\text{Ba}(\text{SO}_4)$	3.85	5.84
Quartz	SiO_2	1.29	0.89
Clinozoisite	$\text{Ca}_2\text{Al}_3(\text{SiO}_4)_3(\text{OH})$	0.08	0.07
Covellite	CuS	0.26	0.34
Chalcopyrite	$\text{CuFe}^{2+}\text{S}_2$	1.57	1.58
Fe-oxide	$\text{Fe}^{3+}_2\text{O}_3$	0.13	0.12
Albite	$\text{NaAlSi}_3\text{O}_8$	0.42	0.27
Galena	PbS	0.23	0.41
Plagioclase	$\text{Na}_{0.5}\text{Ca}_{0.5}\text{Si}_3\text{AlO}_8$	0.12	0.08
Al-oxide	AlO	0.72	0.56
Muscovite	$\text{KAl}_3\text{Si}_3\text{O}_{10}(\text{OH})_{1.9}\text{F}_{0.1}$	0.18	0.11
Chlorite	$(\text{Mg},\text{Fe})_3(\text{Si},\text{Al})_4\text{O}_{10}(\text{OH})_2 \cdot (\text{Mg},\text{Fe})_3(\text{OH})_6$	0.14	0.07
Rutile	TiO_2	0.06	0.04
Calcite	$\text{Ca}(\text{CO}_3)$	0.08	0.04
K-feldspar	KAlSi_3O_8	0.16	0.06
Kaolinite	$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$	0.06	0.04
Chalcocite	CuFe_9S_5	0.46	0.62
Tetrahedrite	$(\text{Cu},\text{Fe})_{12}\text{Sb}_4\text{S}_{13}$	0.01	0.01
Bornite	$\text{Cu}_5\text{Fe}^{2+}\text{S}_4$	0.46	0.58
Cu_Sulphate	CuSO_4	0.02	0.04
Other		0.40	0.40
Total		100.00	100.00

fugacity, etc.), can also be defined as chalcopyrite exsolutions. Chalcopyrite in sphalerite (chalcopyrite disease) is one of the most common associations in metamorphosed sulfide deposits (i.e., below upper amphibolite facies) (Barton and Bethke, 1987; Bente and Doering, 1995; Lockington et al., 2014; Govindarao et al., 2018). Chalcopyrite inclusions in sphalerite are very common and represent, under various circumstances, exsolution and co-precipitation, as well as a replacement (Çiftçi, 2011).

Chalcopyrite disease texture is most common in massive sphalerite-galena-pyrite-chalcopyrite-pyrrhotite \pm Au mineralization (MSGC) and footwall disseminated to semi-massive mineralization (FWD) (Tiu et al., 2021).

Fig. 3 shows the microscope images of polished sections of the feed sample. The data indicate a very low liberation degree between copper minerals and sphalerite. The SEM image, mapping image and EDS results are shown in Fig. 4 and Fig. 5, respectively.

Table 3. Size-by-size liberation of copper minerals, sphalerite and pyrite

Chalcopyrite	Free	Binary Association							Ternary Association								
		Cpy	Chco	Bor	Cov	Pyr	Sph	Bar	Cpy	Chco	Bor	Cov	Pyr	Sph	Bar	Other	Total
75+45 μm	36.21		2.66	3.43	0.97	30.74	2.15	1.44		1.76	3.43	1.68	9.65	1.53	2.88	37.68	100
45+20 μm	42.03		2.18	5.11	0.78	27.62	1.88	2.51		2.31	2.29	1.42	6.41	1.38	2.45	1.63	100
Chalcosite																	
75+45 μm	13.43	22.02		4.05	0.25	29.44	0.78	0.12	7.86		3.23	0.88	11.81	1.11	0.82	4.20	100
45+20 μm	26.52	12.56		8.16	0.01	16.37	0.79	2.13	8.69		6.46	2.37	7.67	1.50	0.43	6.34	100
Bornite																	
75+45 μm	12.96	8.33	0.51		1.22	28.90	0.99	2.06	11.43	2.72		3.21	21.81	1.46	1.03	3.37	100
45+20 μm	19.29	8.09	2.01		3.01	27.06	1.68	1.67	5.45	4.07		2.92	14.91	1.27	2.26	6.31	100
Covellite																	
75+45 μm	38.49	6.73	0.13	1.52		17.68	0.00	0.07	7.67	2.99	7.43		13.84	0.21	0.52	2.72	100
45+20 μm	38.33	6.06	0.00	9.85		17.17	0.00	0.85	4.67	3.72	6.44		8.49	0.01	0.08	4.33	100
Pyrite																	
75+45 μm	94.41	0.96	0.28	0.62	0.17		1.15	1.01	0.24	0.07	0.18	0.06		0.09	0.17	0.59	100
45+20 μm	94.57	1.20	0.14	0.76	0.13		1.07	0.82	0.21	0.07	0.19	0.05		0.08	0.13	0.58	100
Sphalerite																	
75+45 μm	46.01	4.14	0.80	0.14	0.00	29.80		2.53	2.03	0.42	0.69	0.08	5.01		1.81	6.54	100
45+20 μm	55.79	2.58	0.42	2.78	0.00	23.16		2.83	1.39	0.52	0.55	0.00	3.51		1.42	5.05	100

Cpy: Chalcopyrite, Chco: Chalcosite, Bor: Bornite, Cov: Covellite, Pyr: Pyrite, Sph: Sphalerite, Bar: Barite

The distribution areas of the elements sulfur, iron, copper, and zinc overlapped greatly, which showed that the elements iron, copper, and zinc mainly existed in the form of sulfide. However, the distribution of Cu elements was found distributing dispersedly in sulfide ore, with an extremely fine embedded particle size (Fig. 4). In addition, backscattered images, and maps of the composite particles with complex locking texture are also represented in Fig. 6. MLA studies have also shown that the particle size of copper and zinc minerals is fine, and liberation degree is low. It is difficult to achieve high liberation when the grinding fineness is -0.04 mm.

3.2. Sequential flotation response

Effective separation of copper minerals from sphalerite has not been possible due to the low liberation degree, existence of secondary copper minerals, and micron inclusions of a variety of copper minerals. A detailed study about the effects of pyrite texture on flotation performance was recently presented using different ore types from the same copper deposit by Can et al. (2021), who reported that Ore-B contained framboidal and altered pyrite/marcasite and did not respond to the base flotation conditions due to the framboidal and spongy, inclusion-rich, altered pyrite content and relatively high surface oxidation of altered marcasite particles. The authors also reported that high-grade copper concentrates by Cu-Zn bulk flotation method could be produced at acceptable recoveries using Ore-A, which did not contain framboidal and altered pyrite (Can et al., 2021).

However, relatively positive results were obtained from the tests carried out in a slightly alkaline environment (pH: 7.5) and at 20 μm particle size. In the 14-min. copper flotation stage (Cu-rougher + Cu-scavenger), 20 g/t Aero 3418A, 800 g/t NaCN, 2500 g/t ZnSO₄ and 50 g/t MIBC were used. Cu-rougher concentrate containing 9.7% Cu and 3.9% Zn was obtained with 81.6% copper recovery and 46.4% zinc recovery. As a result of this process, only about half of the sphalerite was depressed. Copper scavenger tailings contained 0.45% Cu and 0.93% Zn.

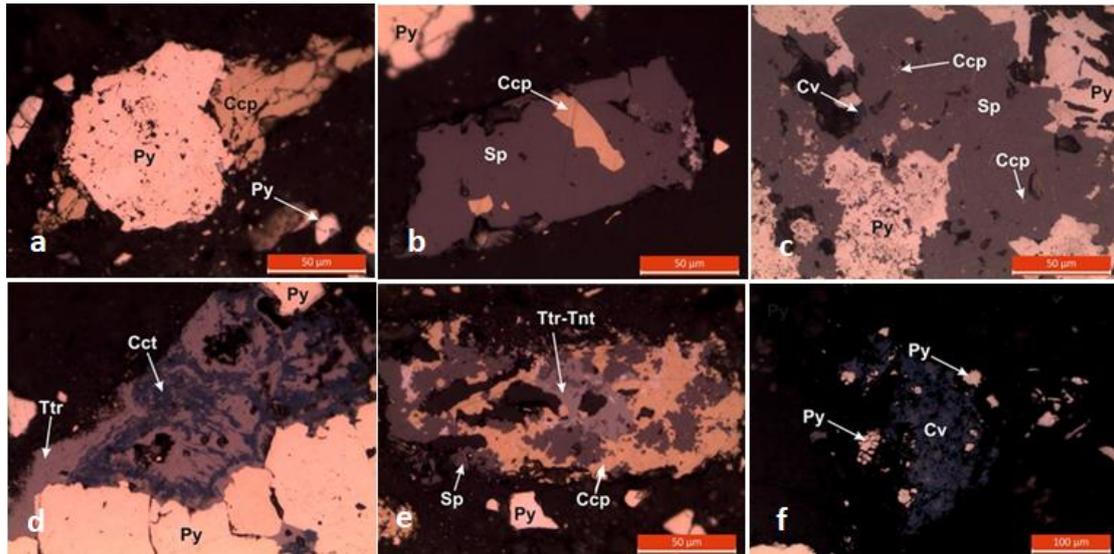


Fig. 3. Photomicrographs of polished sections of sulfide copper-zinc ore. Chalcopyrites developing after pyrite and surrounding pyrite (a), Residual chalcopyrites replaced by sphalerite in sphalerite (b), Very fine-grained chalcopyrite exsolution within the sphalerites developed after the pyrites (chalcopyrite disease) (c), Chalcocite, which surrounds the pyrites and replaces the tetraedrites (d), Combined appearance of tetraedrite-tenantite, chalcopyrite and sphalerite (e), The relationship between chalcocite-covellite and pyrite, which developed by replacing chalcopyrites (f). Ccp – Chalcopyrite; Py – Pyrite; Sp – Sphalerite; Cv – Covellite; Cct – Chalcocite; Ttr – Tetraedrita; Tnt – Tenantite

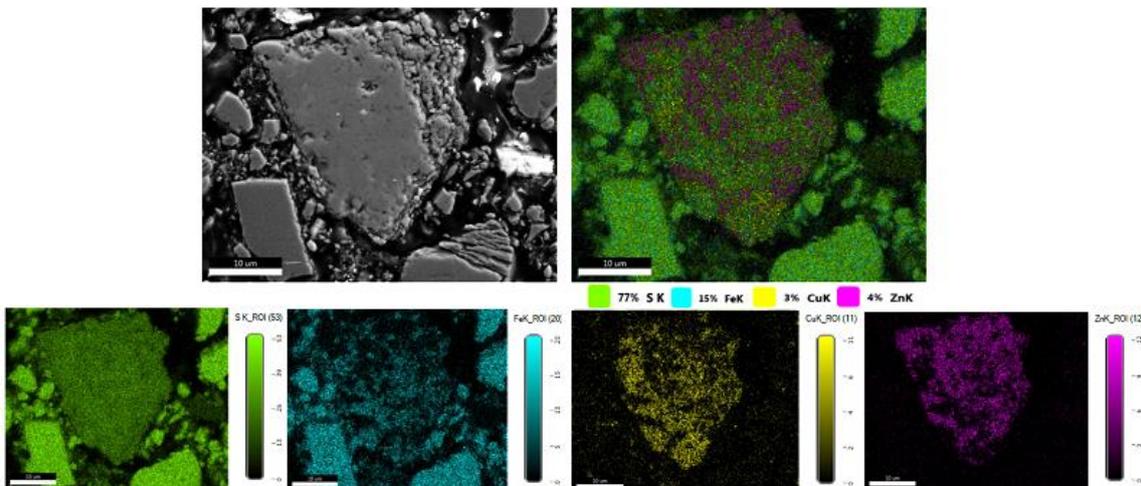


Fig. 4. Backscattered and mapping images and major element distribution map of sulfide Cu-Zn ore

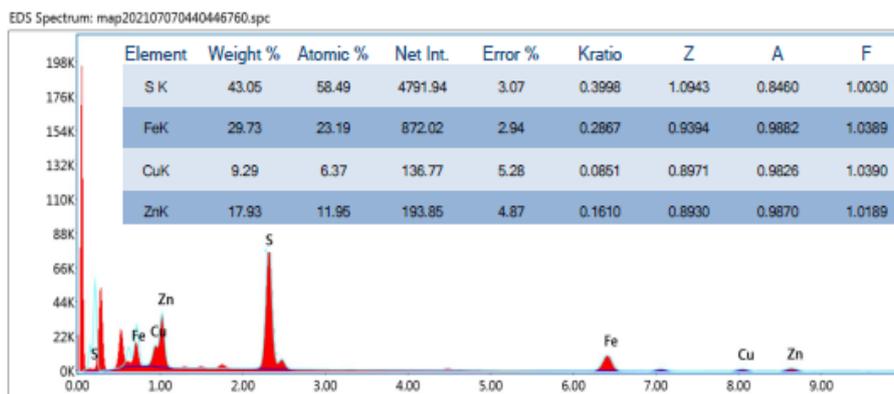


Fig. 5. EDS result of sample

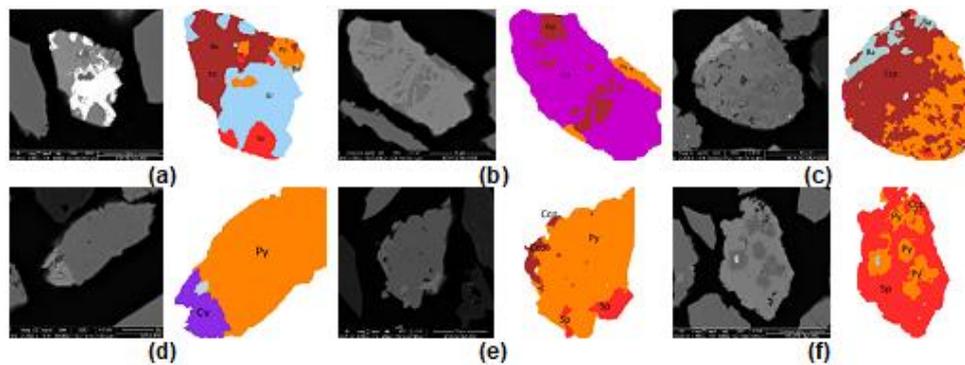


Fig. 6. BSI and composite particles with complex locking texture from MLA. Bornite (a), chalcocite (b), chalcopyrite (c), covellite (d), pyrite (e), and sphalerite (f)

3.3. Copper-zinc bulk flotation

This method utilizes bulk flotation of copper-zinc, while depressing pyrite and other gangue minerals. Cu-Zn bulk flotation conditions were investigated due to the existence of secondary copper minerals, which usually cause the pre-activation of zinc minerals either in situ or during the grinding operation, complex mineralogy of the ore, and the poor selectivity between copper minerals and sphalerite when applying the sequential flotation method.

3.3.1. Effect of particle size

Liberation of valuable minerals from associated gangue minerals is an important and fundamental step in separating an ore mineral from gangue during physical or physicochemical separation processes (Bahrami et al., 2020). A suitable grinding fineness is not only related to the liberation of valuable minerals, but also affects the separation efficiency of valuable minerals in the flotation process (Zhao et al., 2022). The optimal grinding fineness depends on several factors such as ore mineralogy, target mineral(s) and desired recovery rate. In general, the finer the grinding size, the better the mineral liberation, meaning that the mineral particles are separated from the gangue minerals (Sousa et al., 2017). However, excessive grinding can lead to increased production costs due to increased energy consumption, higher reagent consumption, and production of regeneration slimes. Furthermore, fine grinding may also lead to overgrinding, which can negatively impact flotation recovery (Yang et al., 2013), making the selection of appropriate grinding fineness crucial.

The flotation tests were carried out at different particle sizes such as 60, 40, and 25 μm (d_{80} , 80% passing size) to optimize the recovery of copper in the rougher flotation stage. As the grain size decreases, the copper and zinc recovery increases due to the increase in the liberation degree. However, there was no further increase in recovery at smaller particle sizes ($d_{80}=25 \mu\text{m}$) (Fig. 7). Considering the grinding cost, a particle size of $d_{80}:40 \mu\text{m}$ was sufficient for the rougher flotation stage. It is also worth noting that secondary copper minerals, such as covellite, chalcocite, and bornite, are brittle minerals and tend to slime during grinding operations; when secondary copper minerals are found in massive sulfides, they cause a significant separation problem (Bulatovic, 2015). A Cu-Zn bulk concentrate assaying at 13.2% Cu and 9.5% Zn with 84.4% and 88.9% recoveries were achieved at a particle size of 40 μm and a dose of 60 g/t collector (A-3894). Similar results were obtained for 3418A collector. The flotation kinetics of collector A-3894 at 80% passing 25, 40 and 60 μm are shown in Fig. 8.

As shown in Fig. 8, the grades and recoveries of copper slightly decreased with the decrease in the grinding size of 25 μm . As the particle size decreases, the floatability of ore generally decreases gradually due to a number of factors: Increase in the number of particles per unit weight (bubble surface coverage restriction), dwindled inertial force, and changes in surface properties such as surface oxidation or other chemical reactions (Trahar, 1981; Crawford and Ralston, 1988; Miettinen et al., 2010; Agheli et al., 2018). Previous studies in the literature report that most of the copper loss in flotation plants occurs with particle sizes below 20 μm (50%) and above 150 μm (30–40%) (Bulatovic et al., 1998; Barkhordari et al., 2009; Yianatos et al., 2012). Therefore, the optimal grinding particle size of 40 μm (80%) was selected for the rougher flotation stage.

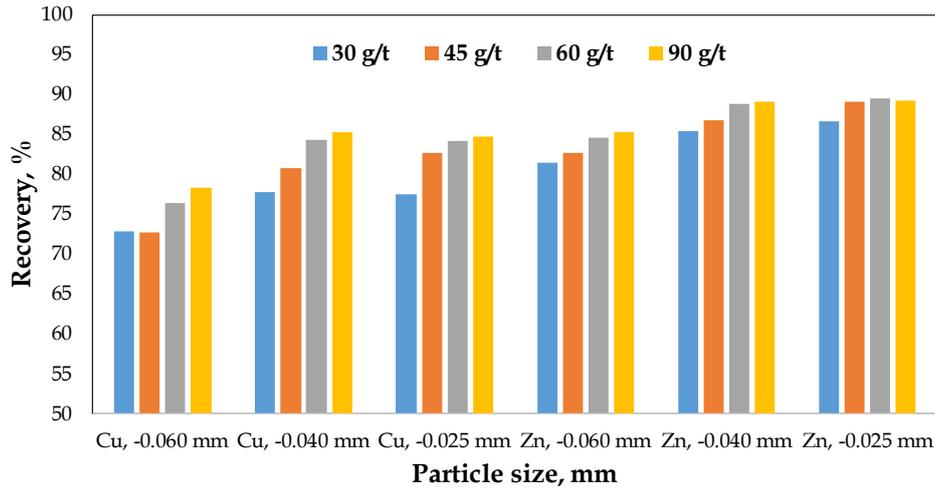


Fig. 7. Effect of particle size on Cu-Zn rougher (bulk) flotation

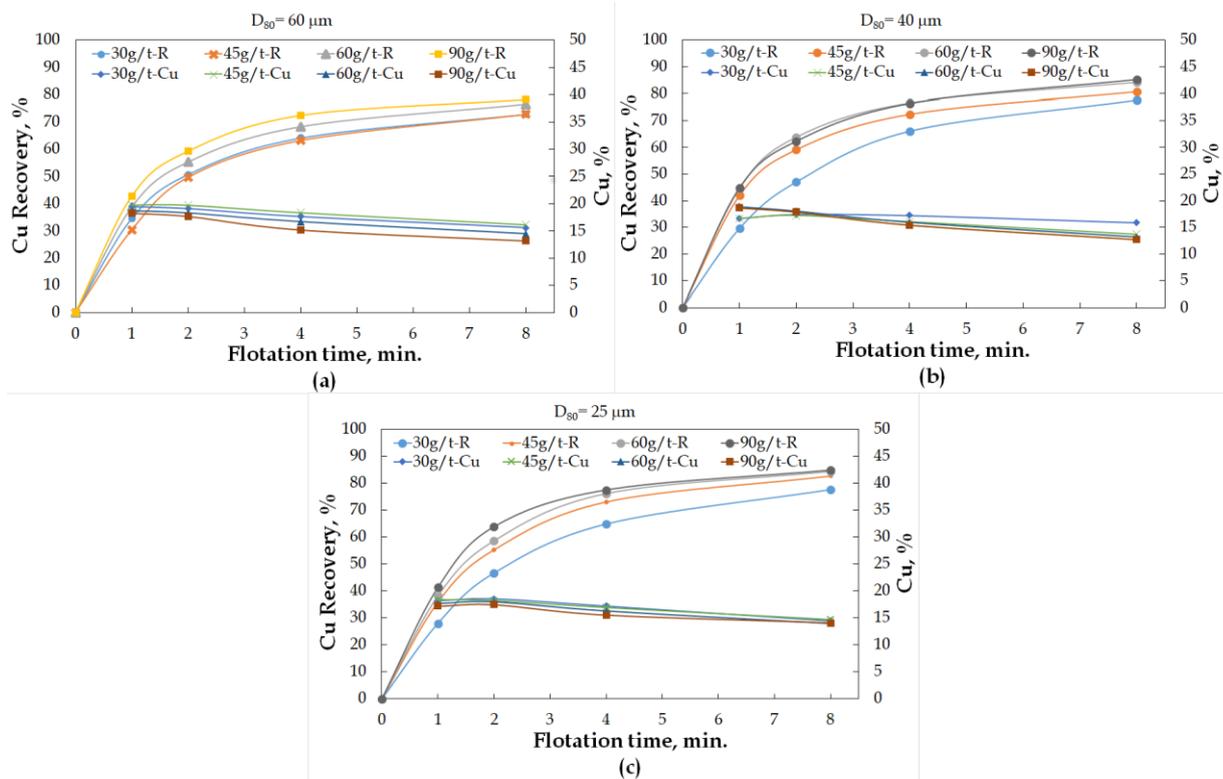


Fig. 8. Kinetic flotation results of Aero 3894 collector (a) 60 μm, (b) 40 μm, and (c) 25 μm

3.3.2. Effect of pH

The flotation behavior of copper-zinc ore sample as a function of slurry pH was investigated for pH 9.5 and 11, and the results obtained from rougher (Ro) + scavenger (Sc) flotation are shown in Table 6. The pH testing was undertaken using different collectors at different dosages. Frother (MIBC) addition was fixed at 50 g/t for rougher flotation and 10 g/t for scavenger flotation. Rougher and scavenger flotation time were kept constant at 8 min and 6 min, respectively. As can be seen from Table 6, high copper recovery is achieved at pH 11 and -40 μm particle size using 60+20 g/t of collector (A-3894 and 3418A). Cumulative copper and zinc recoveries were about 89% and 91%, respectively. When the collector dosage was increased to 90+30 g/t, copper, and zinc recovery slightly increased and reached 89.9% and 91.4%, respectively. Furthermore, 91.6% Cu and 92.5% Zn recoveries were obtained using 3418A at pH 11 and a dosage of 90+30 g/t.

Table 6. Effect of pH on bulk flotation performance for various collectors and their dosages

Collector	Collector dosage, (g/t) (Ro+Sc1+Sc2)	Wt. %	pH=9.5, d ₈₀ =40 μm					
			Grade, %		Recovery, %			
			Cu	Zn	Cu, R	Zn, R	Fe, R	S, R
A-3894	30+20	16.13	10.88	7.61	84.98	88.52	12.97	14.96
	45+20	16.03	11.15	7.62	86.23	88.99	12.87	14.92
	60+20	18.63	9.81	6.57	87.87	90.39	15.60	14.89
AF 238	30+20	12.29	13.14	9.44	77.00	82.02	8.81	10.96
	45+20	14.65	11.82	8.23	82.85	86.00	11.26	13.30
	60+20	14.32	11.97	8.38	83.00	86.97	10.89	13.02
3418A	30+20	13.22	11.98	9.04	76.84	85.69	10.12	11.86
	45+20	14.43	11.94	8.45	82.41	88.24	11.18	13.14
	60+20	16.50	11.17	7.73	84.66	88.94	13.17	15.22
SIPX	60+20	13.66	12.81	8.97	82.51	87.65	10.47	12.45
pH=11, d₈₀=40 μm								
A-3894	30+20	13.93	12.44	8.85	85.19	88.84	10.86	12.69
	45+20	14.30	12.49	8.66	86.69	89.47	11.18	13.35
	60+20	16.53	10.81	7.54	88.80	90.88	13.72	15.73
	90+30	18.47	9.76	6.67	89.89	91.42	15.15	17.26
AF 238	30+20	14.31	12.55	8.48	82.98	87.09	10.78	12.85
	45+20	15.64	11.85	7.75	85.92	89.42	12.22	14.56
	60+20	15.15	11.81	7.87	87.19	89.20	11.96	14.26
3418A	30+20	13.78	12.92	8.63	87.32	89.02	10.88	12.85
	45+20	15.10	11.79	7.93	88.22	89.82	12.27	15.15
	60+20	16.39	11.67	7.67	88.75	90.38	13.69	15.67
	90+30	19.89	9.58	6.50	91.61	92.54	17.22	19.39
SIPX	60+20	16.09	11.61	7.86	87.09	89.33	12.91	14.99
	90+30	17.1	10.71	7.38	88.10	90.10	14.2	16.4
KEX	30+20	13.34	8.61	7.30	55.33	71.40	10.27	11.85
	60+20	18.48	8.77	6.29	77.71	83.59	15.11	17.16
SIPX+KAX	Ro:(45+15) Sc:(20+10)	17.29	10.48	7.27	88.96	90.36	14.46	16.23
	Ro:(60+20) Sc:(20+10)	19.30	9.58	6.55	89.54	91.46	16.56	18.73
	Ro:(90+20) Sc:(20+10)	25.70	7.41	5.06	90.56	92.18	24.81	26.38

3.3.3. Effect of collector type and dosage

Different collector dosages for rougher and scavenger stages (30+20, 45+20, 60+20, and 90+30 g/t) were tested to investigate the effect of collector addition rate at size of d₈₀:40 μm. Impeller speed, air flow rate, and pH during conditioning and flotation in the cell were maintained at constant 1200 rpm, 2.5 dm³/min and 11, respectively, for all experiments. In kinetic rougher flotation tests, four flotation concentrates were collected after cumulative times of 1, 2, 4 and 8 min, and in the scavenger stage, the froth product was collected for 6 min.

Fig. 10 presents the results from bench scale flotation tests of copper-zinc ore using different collectors. In these tests, collector was used at a dose of 60+20 g/t (i.e., 60 g/t for rougher flotation and 20 g/t for scavenger flotation). As can be seen in Fig. 10, there is no great difference in kinetics between the tested collectors. Therefore, maximum copper recovery (89%) was achieved using A-3894 and 3418A. A bulk concentrate assaying 10.8% Cu, 7.5% Zn, 13.7 Fe with 88.8% copper, 90.9% zinc and 13.7% iron recoveries was obtained by using A-3894. Concerning the use of 3418A, a bulk concentrate assaying 11.7% Cu, 7.7% Zn, 13.7 Fe with 88.7% copper, 90.4% zinc, and 13.7% iron recoveries was obtained.

Despite higher collector dosage (Fig. 11), no significant improvement on copper recovery was obtained. Furthermore, 91.6% of copper recovery was achieved when 3418A (90+30 g/t) was used. When two-stage scavenger flotation was applied, the copper recovery increased by about 2 points and reached around 92–93%. However, the mass pull increased approximately 1.5 times compared to one-stage scavenger flotation (Fig. 12).

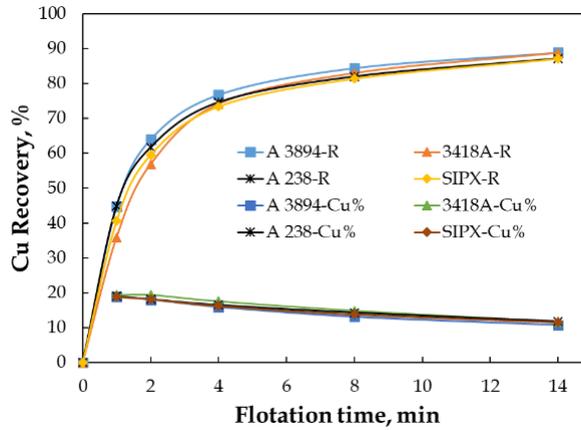


Fig. 10. Kinetic flotation results of different collectors

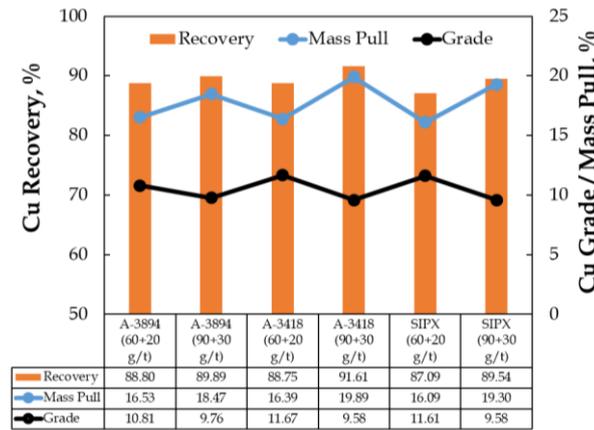


Fig. 11. Effect of collector dosage on recovery and grade

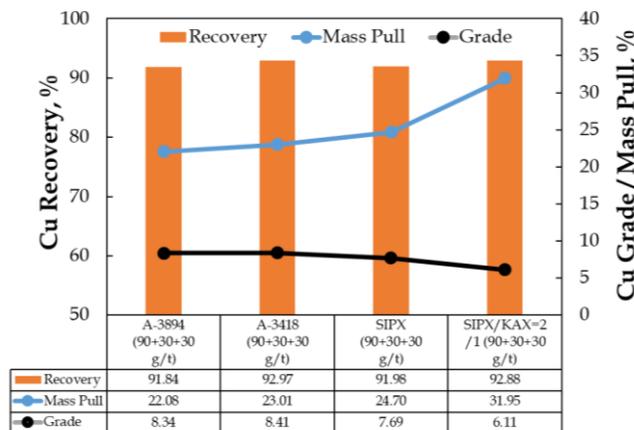


Fig. 12. Results for rougher + two stage scavenger flotation

4. Conclusions

The Cerattepe copper-zinc sulfide ore used in this study was found to have disadvantages, such as complex components, high oxidation rate, fine dissemination, strong association between different

elements, and low liberation degree. Therefore, selective flotation of Cu minerals against sphalerite is impractical. According to the mineralogy results, the major copper-bearing minerals in the ore deposit are chalcopyrite, chalcocite, bornite and covellite. Detailed mineralogical studies revealed that chalcopyrite disease (chalcopyrite inclusions in sphalerite) is commonly present in the ore. Naturally, effective results could not be obtained from the sequential flotation test (separation of copper minerals from sphalerite).

Process mineralogy can be considered as a useful application in mineralogy for the estimation and optimization of an ore process technique. On the other hand, crushing and grinding are energy-intensive processes (especially fine grinding by mill). Therefore, according to the results of size-by-size liberation analysis and the flotation tests, it is sufficient to grind the ore to $d_{80}=40\ \mu\text{m}$ for the Cu-Zn rougher flotation stage. Several types of collectors were tested in batch flotation tests. Similar metallurgical results were obtained by using both thinocarbamate (Aero-3894) and phosphinate (Aerophine 3418A) collectors.

Under optimal conditions for the rougher flotation stage (grain size, $d_{80}=40\ \mu\text{m}$, pH=11, amount of collector reagent 60 g/t (Aero 3894), frother (MIBC)=50 g/t, solid ratio=32%, flotation time=8 min.), a bulk copper-zinc concentrate containing 13% Cu and 9.5% Zn was obtained with 84.4% Cu and 88.9% Zn recovery.

After scavenger flotation, copper and zinc recoveries increased to 88.8% and 90.9%, respectively, with a mass pull of 16.4% using A-3894. When using 3418A, the bulk concentrate assaying 11.7% Cu and 7.7% Zn with 88.7% copper and 90.4% zinc recoveries was obtained.

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