

Polymetallic copper-lead-zinc ores flotation process transforming based on MLA

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Abstract: Sulfide copper ores are commonly accompanied by pyrite, galena, and sphalerite, which creates a substantial difficulty in achieving selective separation during the flotation process. Even with the progress made in flotation techniques, there is an ongoing critical demand for efficient approaches that can reduce the impurity levels in the concentrate. The concentrates obtained from the Xinyuan mine facility were analyzed using AMICS, revealing that the unliberated impurities predominantly contribute to the elevated impurity content in the concentrates, with the similar surface properties of minerals like chalcopyrite, bornite, galena, and sphalerite being of secondary importance. To decrease the presence of liberated or unliberated impurities, the new flotation process includes regrinding and more selective reagents. This new flotation process has effectively increased the recovery rate of copper from 83.33% to 90.32%, while reducing the recovery rates of lead and zinc to 4.72% from 10.56% and to 3.14% from 4.51%, respectively, in copper concentrate. Additionally, it has resulted in an improvement in both the grade and recovery of lead, increasing from 55.57% to 61.14% and from 74.37% to 84.95%, respectively. Likewise, the grade and recovery of zinc have seen an increase, reaching from 52.60% to 55.47% and from 83.73% to 88.16%, respectively.

Keywords: polymetallic sulfide ore, flotation separation, AMICS, process transform

1. Introduction

Due to the swift depletion of high-grade copper, lead, and zinc resources, the exploitation of Copper-Lead-Zinc sulfide ores is gaining increasing attention, particularly in China (Chandra et al., 2009; Xiong et al., 2021). The primary minerals for copper, lead, and zinc are chalcopyrite, galena, and sphalerite, which often coexist as copper-lead-zinc mixed ores in the deposit (Johnson, 2006; Mingli et al., 2006; Quast et al., 2006; Marabini et al., 2007; Liang et al., 2021). These ores are typically associated with low-value sulfides (e.g., FeS₂) and gangue materials (e.g., Al₂O₃, SiO₂), necessitating flotation to separate them and produce individual copper, lead, and zinc concentrates (Marabini et al., 2007; Liu et al., 2008; Ke et al., 2021; Zhang, 2021). However, the flotation separation of Copper-Lead-Zinc sulfide ores is challenging. Copper concentrates obtained through flotation often contain 5-15% lead and 5-10% zinc as impurities, complicating the subsequent smelting process. The high lead and zinc content in copper concentrate can damage the pyrometallurgical process, reduce market value, and lead to the waste of lead and zinc resources (Chandra and Gerson, 2009; Feng, 2020). The motivation of this study is to improve the comprehensive recovery of this kind of resource through a better flotation process (Nagaraj et al., 2016; Chen et al., 2017; Miao et al., 2022).

In recent decades, the field of quantitative and automated mineralogy techniques has undergone significant development, leading to improved accuracy and repeatability in mineralogical measurements. One such technique, the Mineral Liberation Analysis (MLA) system, was developed by the Julius Kruttschnitt Mineral Research Center (JKMRC) in the late 1990s and has gained widespread usage worldwide (Parker et al., 2015; Hu et al., 2021; Wen et al., 2022). By combining high-resolution images,

mineral phase identification, and automatic software analysis, MLA provides precise quantitative mineralogical information. This information is crucial for process design and optimization, as MLA can directly identify mineral phase composition, mode of occurrence, and particle size characteristics, unlike traditional chemical analysis methods. Despite the relatively high cost of MLA tests, its indispensability in minerals engineering is undeniable (A et al., 2019; Tanaka et al., 2021; Wen et al., 2022). Furthermore, advancements in measurement and computer techniques are expected to rapidly reduce the costs of automated mineralogy, enabling its broader application in various fields. Recently, there has been a growing interest in utilizing MLA for flexible applications, such as mineral resource exploration, on-line process monitoring and prediction, as well as processing design and survey. While there have been studies showcasing the effectiveness of MLA, such as Newcombe's research on how a flash flotation circuit can improve gold recovery in refractory ores, to the best of our knowledge, no reports have been published regarding the optimization of the multiscale sphalerite flotation process based on its mineralogical characteristics.

The Xinyuan mine plant, situated in Sichuan Province, China, processes around 140 tons of ores per hour, with chalcopyrite, galena, and sphalerite constituting the primary valuable minerals in the ore. The plant encounters challenges in effectively treating its complex polymetallic copper-lead-zinc ores, as the copper concentrate contains significant portions of galena and sphalerite, and the lead concentrate contains sphalerite, thereby impacting the grade and recovery of chalcopyrite, galena, and sphalerite. To rectify these issues, MLA was employed to assess the current flotation process, leading to the proposition of a new flotation process aimed at enhancing flotation efficiency.

2. Materials and methods

2.1 Materials

Fig.1 shows the original flotation process used in Xinyuan mine plant. The flotation process in the plant consists of three parts: copper flotation, lead flotation and zinc flotation, which beneficiates three valuable metals (i.e., copper, lead and zinc). Each flotation process includes one roughing, three cleaning, and three scavenging operations. Copper, lead and zinc concentrates were characterized by MLA for process defects inspection, in which samples were obtained in original process. Next, a new flotation process was made to improve separation performance, and corresponding samples were collected and characterized. BK905 and BK906 are commonly used collectors, and they were developed by mining and Metallurgy Technology Group Co., Ltd. The main component of BK905 and BK906 are thionocarbamate and aniline aerofloat, respectively. Collector EM602, depressants EMY30 and EMT-42 are developed by Institute of Multi-purpose Utilization of Mineral Resources. The main component of EM602, EMY30 and EMT-42 are fulvic acid, acrylonitrile butyl xanthate and sulphate. The main component of frother 2# oil is terpineol. Additionally, flotation agents such as butyl xanthate, ZnSO_4 , Na_2SO_3 , Na_2S , CaO , CuSO_4 were also used. All the mentioned reagents are of industrial grade.

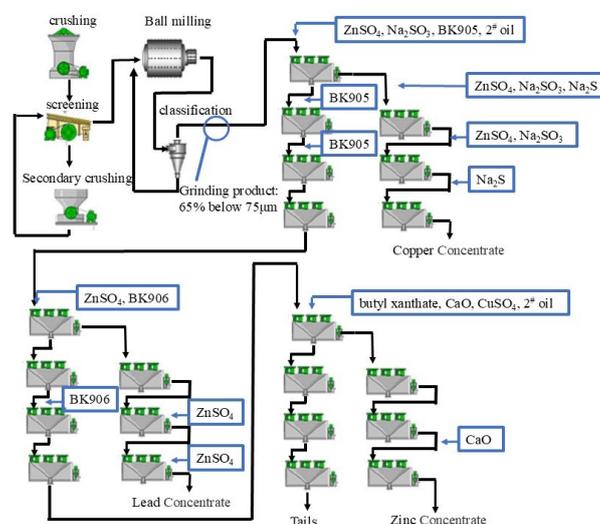


Fig.1. Original flotation process

2.2. AMICS analysis

The AMICS (Advanced Mineral Identification and Characterization System) is a powerful tool for analyzing mineral composition, content, particle size distribution, and intergrowth. This paper uses the AMICS to analyze the froth flotation products. For efficient and accurate testing, homogeneous samples of 100g are prepared and screened using standard sieves with 53 μm and 30 μm mesh sizes. The three granular samples obtained after screening are mixed with epoxy resin and hardener, and the cured target samples are polished and carbon sprayed to ensure their conductivity. The AMICS test is performed using the German ZEISS Sigma 500 Automated Mineral Analyzer, with SEM Control and AMICS Investigator software controlling the instrument's settings and image acquisition. The X-ray mode (BSE, XBSE) is used to test the samples during the analysis period, with a working distance of 10.82 mm, a measurement accuracy of 0.56 μm per pixel, a probe current of 10 nA, and an electron beam acceleration voltage of 20 kV. The BSE image grayscale value calibration is set using epoxy resin as the background (BSE grayscale value > 255). AMICS Process software is used to process the test data.

2.3. Flotation experiments

To determine the optimal conditions for the flotation circuit test, experiments were conducted to determine the optimal regrind particle size and reagent dosage. The regrind particle size experiment involved using a rod mill (XMB- Φ 200 \times 240) to grind the ore to different particle sizes, aiming to identify the optimal regrind particle size. Fig. 2. Show the process flowsheet adopted for regrinding fineness flotation. The flotation tests were performed using a single slot flotation machine (XFD of 0.5 L, 1 L, and 3 L). After determining the optimal reagent dosage, the closed-circuit experiments were conducted to evaluate the performance of the new process. The closed-circuit experiment involved mixing 1 kg of the original ore with 2 L of tap water and stirring the mixture. The final concentrates and tailings were filtered, dried, and weighed, for grade analysis. The copper, lead, and zinc recoveries were calculated based on the weight and grade of concentrates.

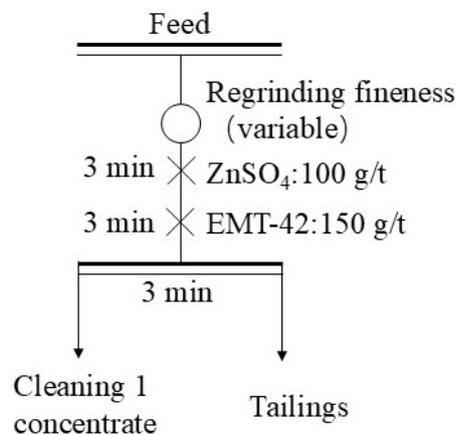


Fig. 2. Technological process for regrinding fineness flotation

3. Results and discussion

3.1 Original process survey

Mineral composition of copper, lead and zinc concentrate were shown in table 1. As can be seen, there were 35.87% of tetrahedrite, 15.56% of Chalcopyrite, 1.89% of bornite in copper concentrate, all of them were copper bearing minerals. It is worth noting that some impurities like galena, sphalerite, pyrite in copper concentrate, sphalerite, pyrite in lead concentrate and of gangue minerals, pyrite in zinc concentrate, which need to be removed during flotation to avoid lowering the concentrate grade. Therefore, the aim of the research presented in this paper is finding a way to decrease the contaminant content in copper, lead and zinc concentrate.

MLA was utilized to characterize the concentrates collected from the original process to some information regarding the mineral liberation. Fig. 3 presents the mineral composition maps of copper

concentrate, lead concentrate and zinc concentrate. As shown in Fig. 3 (a), the content of gangue minerals in the copper concentrate significantly decreased, while minerals such as bornite, chalcopyrite, and pyrite were enriched, demonstrating the selectivity of the flotation process. As can be seen in fig. 3., there were still some intergrowth of pyrite/ sphalerite / galena. The main minerals in Figs. 3b and 3c are galena and sphalerite, respectively. We can also find some symbionts in lead concentrate and zinc concentrate, in which the symbionts include continuous mineral facies of sphalerite, pyrite and galena.

Table 1. Mineral composition of copper, lead and zinc concentrate

copper concentrate	minerals	Tetrahedrite	Galena	Chalcopyrite	Sphalerite	Pyrite
	content(wt%)	35.87	17.48	15.56	13.82	13.50
	minerals	Bornite	Gangue minerals	Pyrrhotite	Arsenopyrite	
	content(wt%)	1.89	1.74	0.11	0.03	
lead concentrate	Minerals	Galena	Sphalerite	Pyrite	Gangue minerals	Chalcopyrite
	content(wt%)	65.23	18.65	13.71	1.33	0.51
	Minerals	Tetrahedrite	Pyrrhotite	Bornite	Arsenopyrite	
	content(wt%)	0.32	0.12	0.1	0.03	
zinc concentrate	minerals	Sphalerite	Gangue minerals	Pyrite	Galena	Chalcopyrite
	content(wt%)	79.94	7.68	7.48	3.59	0.96
	minerals	Tetrahedrite	Pyrrhotite	Bornite	Arsenopyrite	
	content (wt%)	0.24	0.08	0.02	0.01	

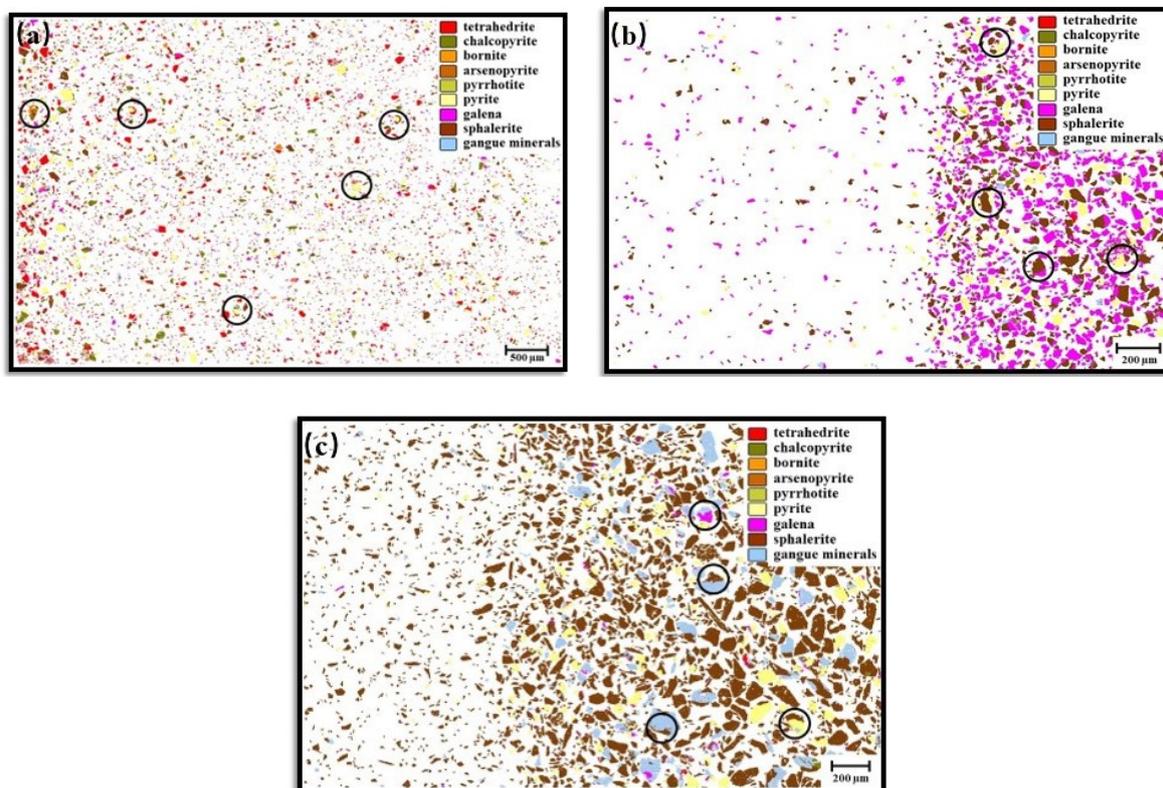


Fig. 3. Mineral composition maps of copper concentrate (a), lead concentrate (b) and zinc concentrate (c)

Table 2. Liberation degree of minerals in copper, lead and zinc concentrate

Minerals in copper concentrate	Liberated mineral percentage/%	Content of symbionts/%								
		Chalcopyrite	Pyrite	Bornite	Arsenopyrite	Pyrrhotite	Gangue minerals	Galena	Sphalerite	Tetrahedrite
Chalcopyrite	88.17	0.00	1.71	0.31	0.06	0.02	0.75	3.66	2.98	2.34
Pyrite	85.09	2.34	0.00	0.82	0.00	1.81	0.59	3.05	3.18	3.12
Bornite	80.53	2.92	5.25	0.00	0.00	0.03	0.84	2.73	2.28	5.42
Arsenopyrite	57.39	21.58	0.70	0.00	0.00	0.00	0.00	5.41	2.65	12.27
Pyrrhotite	30.49	1.27	64.61	0.15	0.00	0.00	0.60	0.15	1.57	1.16
Gangue minerals	82.68	3.35	1.79	0.40	0.00	0.05	0.00	1.88	4.42	5.43
Galena	78.91	4.22	2.39	0.33	0.02	0.00	0.48	0.00	6.24	7.41
Sphalerite	81.36	2.96	2.15	0.24	0.01	0.03	0.98	5.40	0.00	6.87
Tetrahedrite	88.86	1.34	1.21	0.33	0.02	0.01	0.69	3.66	3.88	0.00
Minerals in lead concentrate	Liberated mineral percentage/%	Content of symbionts/%								
		Chalcopyrite	Pyrite	Bornite	Arsenopyrite	Pyrrhotite	Gangue minerals	Galena	Sphalerite	Tetrahedrite
Chalcopyrite	75.06	0.00	1.45	0.00	0.00	0.72	18.00	4.53	0.24	0.00
Pyrite	86.92	0.08	0	0.01	0.49	0.58	9.40	2.42	0.10	0.00
Bornite	88.62	0.00	2.59	0.00	0.00	0.00	6.33	2.46	0.00	0.00
Arsenopyrite	66.08	0.00	26.45	0.00	0.00	0.99	5.56	0.92	0.00	0.00
Pyrrhotite	83.98	0.12	1.72	0.00	0.05	0.00	9.88	4.00	0.25	0.00
Gangue minerals	88.98	0.25	2.32	0.00	0.03	0.82	0.00	7.49	0.11	0.00
Galena	85.87	0.11	0.99	0.00	0.01	0.55	12.38	0.00	0.09	0.00
Sphalerite	67.85	0.50	3.88	0.00	0.00	3.10	16.69	7.98	0.00	0.00
Minerals in zinc concentrate	Liberated mineral percentage/%	Content of symbionts/%								
		Chalcopyrite	Pyrite	Bornite	Arsenopyrite	Pyrrhotite	Gangue minerals	Galena	Sphalerite	Tetrahedrite
Chalcopyrite	61.77	0.00	5.6	0.00	0.00	0.15	4.55	7.79	19.65	0.49
Pyrite	77.18	0.57	0.00	0.06	0.01	2.31	2.28	3.83	13.32	0.44
Bornite	43.92	0.00	21.51	0.00	0.00	0.00	12.85	13.24	2.99	5.49
Arsenopyrite	71.87	0.00	6.34	0.00	0.00	0.00	12.07	4.79	4.93	0.00
Pyrrhotite	27.24	0.45	68.19	0.00	0.00	0.00	0.27	0.61	3.21	0.03
Gangue minerals	85.78	0.21	1.04	0.02	0.01	0.00	0.00	2.05	10.73	0.16
Galena	42.25	2.04	9.83	0.09	0.02	0.05	11.53	0.00	33.39	0.80
Sphalerite	94.00	0.23	1.50	0.00	0.00	0.01	2.63	1.46	0.00	0.17
Tetrahedrite	40.51	1.10	9.70	0.32	0.00	0.02	7.73	6.89	33.73	0.00

3.2. Effect of regrinding

Fig. 4. shows the recovery of copper, lead and zinc as a function of fineness of regrinding product in rough flotation concentrate. The copper recovery of concentrate increased from 84.50% to 91.47%, and then decreased. The lead and zinc recovery decreased from 30.78% to 23.12% and from 22.34% to 10.42%, respectively. The optimum fineness in regrinding is 78% below 45 microns.

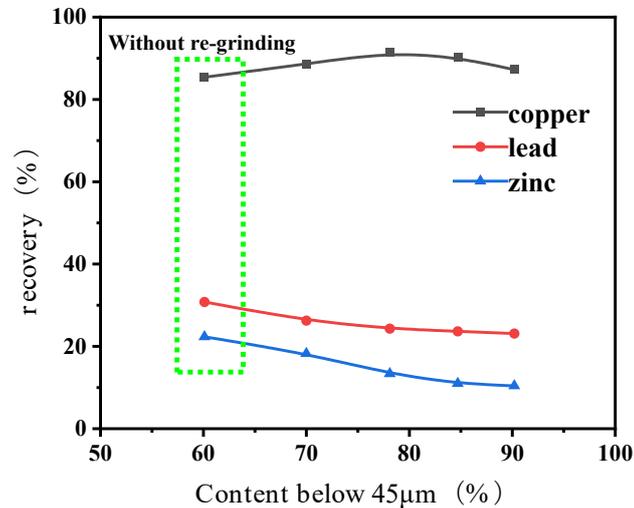


Fig. 4. Recovery of copper, lead and zinc as a function of regrinding fineness

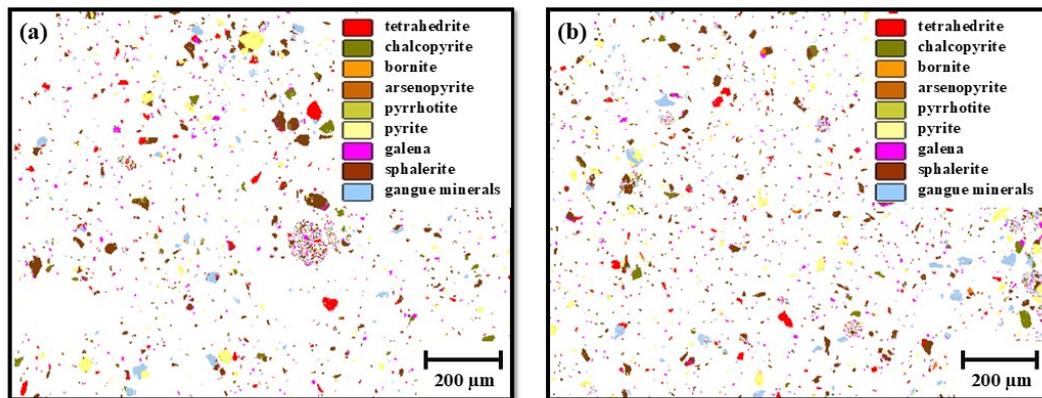


Fig. 5. Mineral composition maps of copper rougher concentrate, (a): before regrinding, (b): after regrinding

Fig. 5a and b show the mineral composition maps of copper rougher concentrate before and after regrinding, respectively. The main minerals in fig.5a and b is tetrahedrite, chalcopyrite, bornite, galena, sphalerite. Intergrowth of pyrite/ sphalerite/ galena in copper concentrate still can be seen in Fig. 5a and b. As can be seen in fig.6, the liberation degree after regrinding is higher than before. This indicates that regrinding can liberate single minerals from intergrowth. Moreover, from copper rougher concentrate to copper concentrate (Table 2), the liberation degree of Cu-bearing minerals (tetrahedrite, chalcopyrite, bornite) is getting higher, pyrite, galena and sphalerite change slightly, but other minerals (tailings, pyrrhotite) are getting lower, which proves the liberated Cu-bearing minerals have higher affinity to be floated into froth product.

3.3 Rough flotation experiments

In rough flotation experiments, $ZnSO_4$ and 2# oil act as a depressant and frother, respectively. Fig. 7(a) demonstrates that as the EMT-42 dosage rises from 400 g/t to 1000 g/t, the grades of copper and zinc increase slightly from 6.39% to 7.24% and 6.14% to 6.84%, respectively, while the lead grade sharply declines from 17.03% to 11.03%. Concurrently, copper and zinc recoveries improve from 79.04% to 84.29%

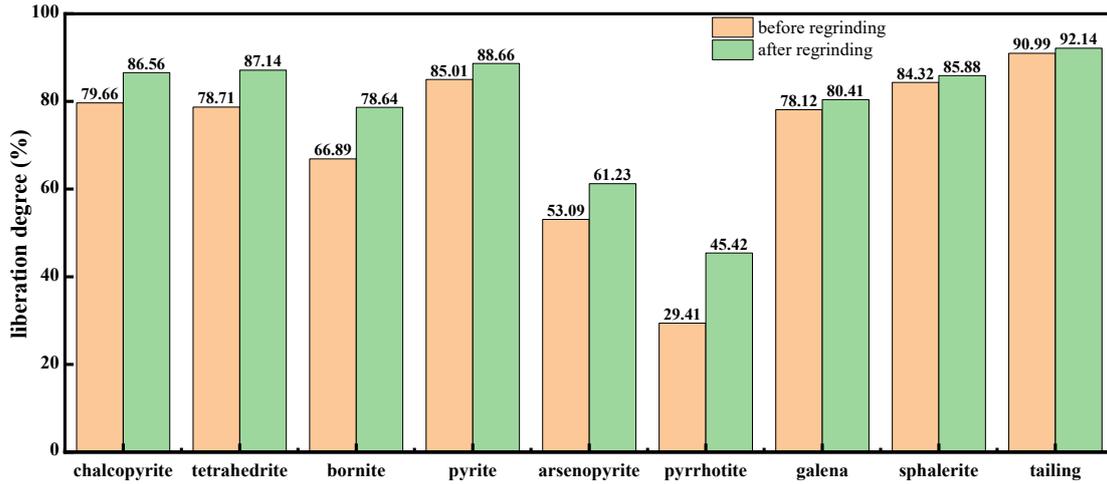


Fig. 6. Mineral liberation degree of copper rougher concentrate before and after regrinding

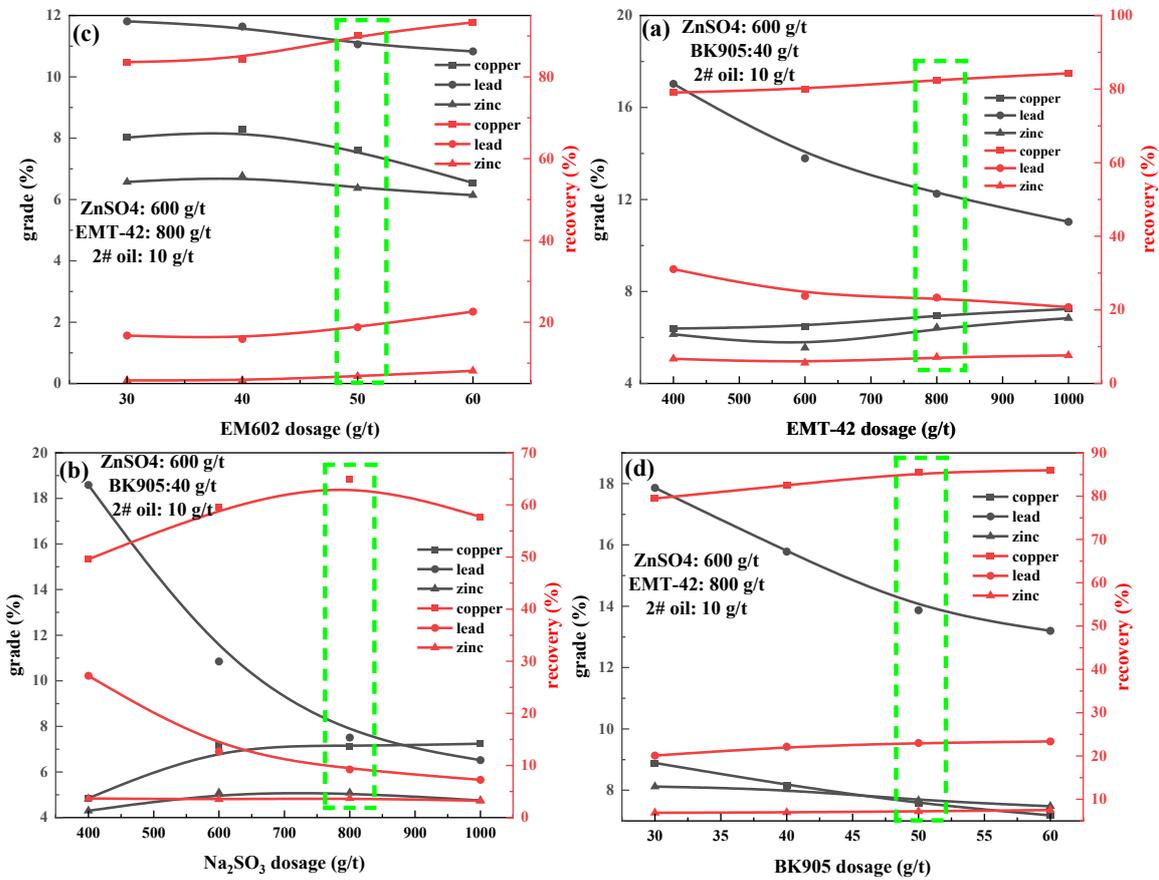


Fig. 7. Recovery and grade of copper, lead and zinc of concentrate as a function of (a) EMT-42, (b) Na₂SO₃, (c) EM602 and (d) BK905 dosages

and 6.70% to 7.62%, respectively, whereas lead recovery drops significantly from 31.07% to 20.75%. The optimal EMT-42 dosage is determined to be 800 g/t, achieving recoveries of 82.51% for copper, 23.24% for lead, and 7.15% for zinc, alongside corresponding grades of 6.97%, 12.25%, and 6.43%, respectively. When Na₂SO₃ is employed as a depressant, the maximum copper recovery reaches only 64.97%, markedly lower than that achieved with the optimal EMT-42 dosage. For Figs. 7(c) and 7(d), EMT-42 is maintained at 800 g/t. Fig. 7(c) further reveals that increasing EM602 dosage reduces copper, lead, and zinc grades from 8.02% to 6.54%, 11.81% to 10.83%, and 6.57% to 6.14%, respectively, while their recoveries

rise consistently – copper from 83.59% to 93.33%, lead from 16.73% to 22.59%, and zinc from 5.76% to 8.11%. The optimal EM602 dosage is 50 g/t. When BK905 was used as collector, the maximum recovery of copper is 85.99%, lower than that achieved with the optimal EM602 dosage (90.18%). The optimum experimental conditions were determined as follows: EMT-42 and EM602 dosages of 800 g/t and 50 g/t, respectively.

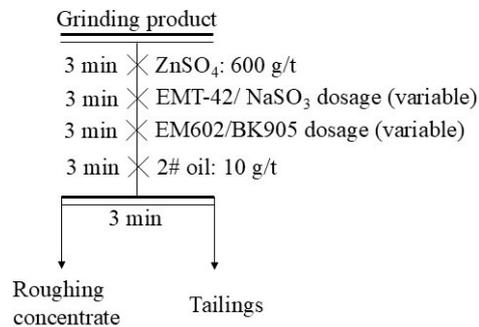


Fig. 8. Technological processes for rough flotation experiments

3.4. Flotation process transform

The original flotation process and reagents fail to effectively separate valuable minerals from tailings. On one hand, collectors BK905 and BK906 in original flotation process has poor selectivity. On the other hand, both copper and lead concentrates contain partially intergrown minerals that are difficult to separate though flotation. To address these issues, a regrinding process has been added to the copper rougher concentrate to reduce intergrown minerals in the copper concentrate. Additionally, the original three scavenging has been changed to four scavenging. The flotation reagent system has been optimized using more selective reagents, EMT-42, EMY-30, and EM602, to better separate chalcopyrite, galena, and sphalerite. The new flotation process and reagent system are shown in Fig. 9.

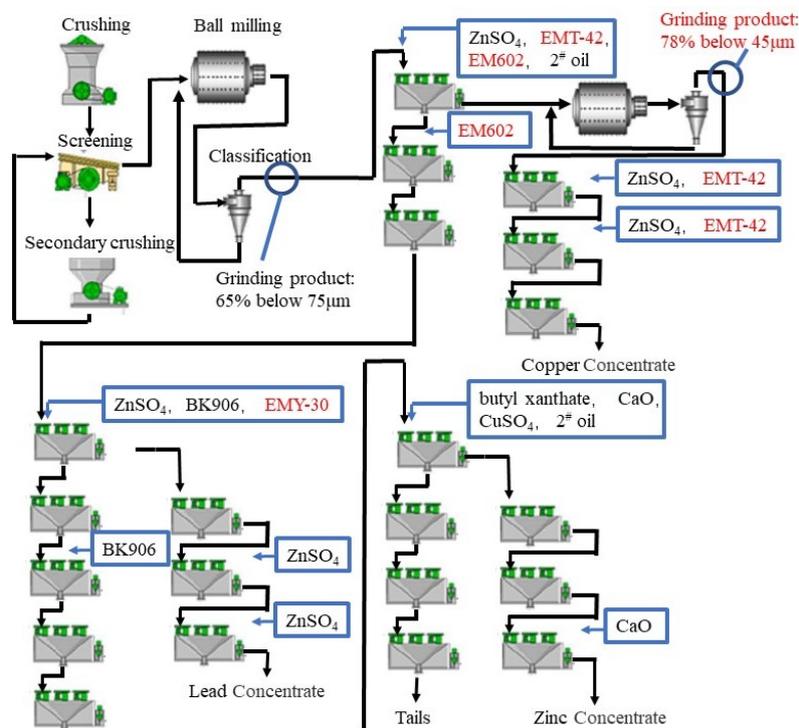


Fig. 9. new flotation flowchart

3.5. Closed circuit experiment

Closed-circuit tests were performed according to the flowchart shown in Fig. 10, and the results are presented in Table 3. In new flotation process, copper concentrate had a slightly lower Cu grade

(20.45%), much higher Cu recovery (90.32%), lower grade and recovery of Pb and Zn. Similar trends were noted in the lead and zinc concentrates. The lead concentrate showcased an increased lead grade and recovery, reaching 61.14% and 84.95% respectively. However, the grade and recovery of copper and zinc were found to be decreased in comparison to the original flotation process. In the case of the zinc concentrate, there was a notable increase in zinc grade and recovery, recorded at 54.47% and 88.16% respectively, yet the grades and recoveries of copper and lead were comparatively lower than those achieved in the original flotation process. Closed-circuit flotation experiment results indicates that new flotation process can increase Cu, Pb and Zn recovery in copper, lead and zinc concentrate, respectively, without decrease their grade.

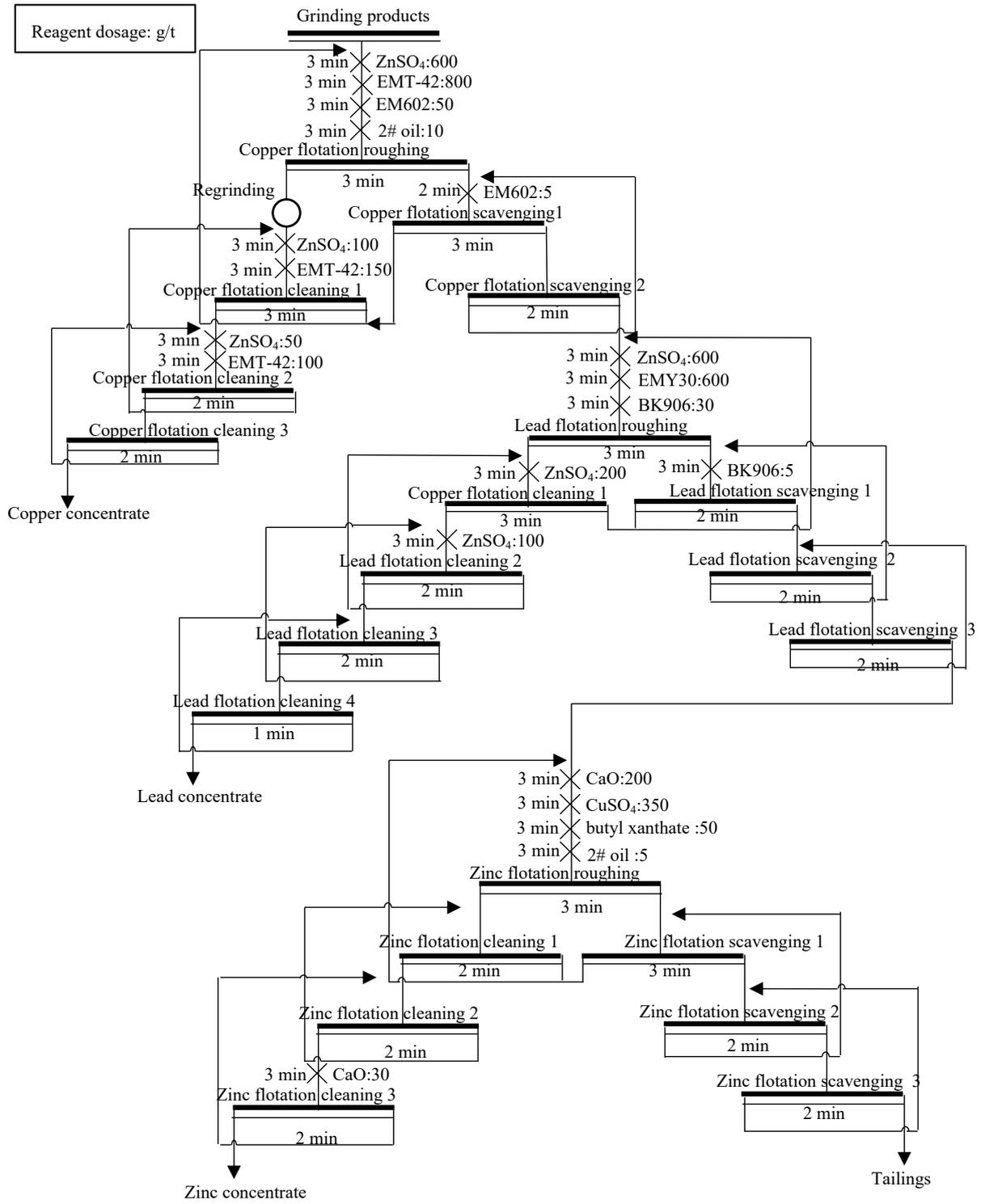


Fig. 10. Closed-circuit flotation flowsheet

Table 3. closed-circuit flotation experiments of original and new process

samples	products	grade (%)			recovery (%)		
		Cu	Pb	Zn	Cu	Pb	Zn
original flotation process	copper concentrate	20.62	15.14	11.27	83.33	10.56	4.51
	lead concentrate	0.38	55.57	12.31	2.95	74.37	9.44
	zinc concentrate	0.46	3.07	52.60	7.40	8.53	83.73
	tail	0.035	0.21	0.13	6.32	6.54	2.32
new flotation process	copper concentrate	20.45	13.58	9.07	90.32	4.72	3.14
	lead concentrate	0.17	61.14	5.02	1.40	84.95	4.65
	zinc concentrate	0.14	1.13	55.47	2.53	1.11	88.16
	tail	0.02	0.21	0.16	5.75	9.22	4.05

4. Conclusions

In this study, AMICS was used to analyze copper, lead and zinc concentrate in Xinyuan mine plant as well as rougher concentrate before and after regrinding. Moreover, closed-circuit flotation test was used to verify the effect of new flotation process. The main conclusions obtained from experimental tests are as follows:

- (1) The majority of impurities present in the concentrates of copper, lead, and zinc have not been liberated. This indicates that intergrowth are the primary contributors to the high impurity grades in the concentrates, with the similar surface characteristics of minerals such as tetrahedrite, chalcopyrite, bornite, galena, and sphalerite playing a secondary role.
- (2) Regrinding can increase the minerals liberation degree in copper rougher concentrate. The new flotation process incorporates regrinding and more selective flotation reagents to reduce the presence of liberated impurities and intergrowth. As a result, this improved flotation process has successfully increased copper recovery rates from 83.33% to 90.32% and decreased the recoveries of lead and zinc from 10.56% to 4.72% and from 4.51% to 3.14% in the copper concentrate. Furthermore, it has led to an increase in both the grade and recovery of lead, raising from 55.57% to 61.14% and from 74.37% to 84.95%, respectively. Similarly, the grade and recovery of zinc have been elevated from 52.60% to 55.47% and from 83.73% to 88.16%, respectively.

Acknowledgments

The authors would like to thank anonymous reviewers for their remarks and comments that improved an earlier version of the manuscript.

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