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Optimizing Pb-Ag Recovery Rate in the Flotation Process of Polymetallic Ores from the Northern Andean Region of Peru through Mineralogical Characterization

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ABSTRACT

A mineralogical characterization study to optimize lead-silver (Pb-Ag) recovery rate by flotation was conducted at three adits named Esmeralda, Orión, and Hércules. The variables investigated included the dosage of the reagent blend (AP-3418/AR-404), AR-242, and pH. At the Esmeralda adit, a recovery of 81.99% lead at a concentrate grade of 58.66% and 83.79% silver at a head grade of 310.64 oz/t was achieved using a dosage of 18 g/t reagent blend (AP-3418/AR-404), 5 g/t AR-242, and an optimum pH value of 11.5. At the Orión adit, a recovery of 81.71% lead at a concentrate grade of 61.6%, and 81.24% silver at a head grade of 85.39 oz/t were achieved using a dosage of 30 g/t reagent blend (AP-3418/AR-404), 5 g/t AR-242, and an optimum pH value of 10.5. Finally, at the Hércules adit, a recovery of 82.4% lead at a concentrate grade of 54.12%, and 80.88% silver at a head grade of 62.63 oz/t grade were achieved using a dosage of 30 g/t reagent blend (AP-3418/AR-404), 15 g/t AR-242, and a pH value of 11.5.

Keywords: characterization, Minitab, adit, flotation.

INTRODUCTION

Until the end of 2019, Peru ranked as the second-largest silver producer and the third-largest lead producer in the world, according to a report by the United States Geological Survey (2019). However, from 2020 onwards, there has been a decline in the production of these metals. As a result, Peru has dropped to the third and fourth positions for silver and lead production worldwide, respectively, surpassed by China and the United States, according to figures reported by the Peruvian Statistical Mining Bulletin (Ministerio de Energía y Minas, 2022).

The lead content of natural galena is generally low, requiring concentration to obtain a commercial product (Yekeler & Yekeler, 2006). Flotation has been the conventional method for recovering polymetallic ores, with low production costs and low environmental impact. As a result, it remains irreplaceable in the processing of sulfide ore resources (Xie et al., 2021). Notably, about 95% of global lead production comes from its sulfide species (Zou et al., 2022). The success of the flotation process can be attributed to the ability

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of the chemical reagents to form stable complexes with metal ions in aqueous solutions or on ore surfaces. This enhances the physical and chemical properties of the surface, allowing for the selective separation of sulfide species from the gangue (Chen, 2021). Dithiophosphoric acid compounds have been developed as flotation collectors for the sulfide species of lead, among them, dithiophosphate (DTP) and dithiophosphinate (DTPI) are the most widely used due to their high selectivity in galena concentration (Tercero et al., 2019). Concentrating galena with xanthate-type collectors is also possible, which do not hydrolyze, oxidize, or decompose into other species, unlike dithiophosphates (Elizondo et al., 2021; Shen et al., 2016).

Silver is often found as a trace or minor component in galena; as a result, less attention is often given to silver recovery compared to the main component (Ayllón, 2013; Aranda, 2014; Nassar et al., 2015; Song et al., 2021). However, the significance of silver has grown due to its role in the development of green technologies, such as solar panels and hybrid vehicles. This has made optimizing silver production during the flotation process critical for the mining and metallurgical sectors (Tiu et al., 2021). Furthermore, silver is recognized as one of the essential materials needed to achieve the Sustainable Development Goals (SDGs) set for 2030 (Dou et al., 2023).

Astucuri (1994) discusses the intricate physical and chemical characteristics of certain species, emphasizing the need for a thorough geological characterization of the deposits that contain them. This process involves identifying and quantifying mineral species, their volumetric distribution, degrees of liberation, and the associations in the ore; it also includes analyzing rock structures associated with the deposit (Yovanovic, 2004; Melgarejo et al., 2010; Bertolino et al., 2014; Ramos & Orihuela, 2017; Taya, 2018). Such detailed information is crucial for assessing the impact of the species involved in mineral processing; it aids in effectively segmenting the deposit and optimizing the recovery of valuable species (López & Ipanaqué, 2008; Bustamante et al., 2008; Ojeda et al., 2010; Espinoza et al., 2021).

Among the conventional techniques used for the characterization of mineral species are X-ray powder diffraction (XRD), quantitative X-ray diffraction, scanning electron microscopy and energy dispersive X-ray spectroscopy (SEM-EDS), cathodoluminescence, and electron microprobe (EMP). These are widely used due to their relatively low cost. In contrast, non-conventional techniques such as particle-induced X-ray emission (Micro-PIXE), secondary ion mass spectrometry (SIMS), and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) are less accessible and significantly more expensive (Melgarejo et al., 2010; Alves, 2014).

Song et al. (2021) investigated the flotation of silver-bearing galena using collectors such as dibutyl ammonium dithiophosphate (ADD), ethyl xanthate (EX), and diethyldithiocarbamate (DDC). They found that silver-bearing galena is more effectively floated at a pH level of 9.5. The results indicated that the ADD collector yielded better recovery rates than both the EX and DDC collectors. The authors conducted micro-flotation experiments on 2 g samples ground to 100% -200 mesh. They also performed conventional flotation tests in a 1500 ml flotation cell using 500 g of raw ore ground to 80% -74 microns, an agitation rate of 1500 rpm, and an airflow rate of 20 L/min. To further investigate the behavior of silver-bearing galena, the authors conducted simulations based on the density functional theory (DFT) method. The best flotation tests using the ADD collector achieved a recovery rate of 85% for galena and over 90% for silver-bearing galena.

This research paper is relevant because it uses experimental designs and analysis of variance to determine the optimal dosage of reagents for maximizing the recovery rate and quality of Pb-Ag concentrate. The calculated reagent dosages aim to predict recovery percentages and quality in polymetallic deposits located in the northern Andean region of Peru. Additionally, this research contributes to the improvement and optimization of outcomes in Pb and Ag mining, while also enhancing the academic understanding related to their extraction.

General Objective

Optimizing lead-silver recovery rate in the flotation process of polymetallic ores from the Andean region of Peru through mineralogical characterization and experimental designs.

Specific Objectives

- Identify the sample components, degrees of liberation, and the presence of free and interlocked grains from the Esmeralda, Orión, and Hércules adits in the Andean region of Peru.

- Determine the influence of the reagent blend (AP-3418/AR-404) dosage, AR-242, and the pH of the pulp, along with their contributions to the flotation process using Plackett-Burman experimental designs and Minitab statistical software.
- Optimize the lead and silver recovery rate in the flotation process of polymetallic ores from three adits located in the Andean region of Peru.

General Hypothesis

Mineralogical characterization and experimental designs optimize the lead-silver recovery rate by the flotation process of polymetallic ores in the Andean region of Peru.

Specific Hypotheses

- Mineralogical characterization allows for the identification of the mineral species present in a sample, as well as the degrees of liberation and bonds found in various mineralogical formations.
- Analyzing the interaction of reagent blend (AP-3418/AR-404) dosages, AR-242, and the pH of the pulp using Plackett-Burman experimental designs in Minitab statistical software helps determine the extent of their contribution to the flotation process in various adits.
- Optimizing the lead and silver recovery rate by flotation leads to improved metallurgical performance.

Theoretical Justification

The optimization of reagent dosage and pulp alkalinity in the flotation process enhances the metallurgical performance of lead-silver concentrates, thereby improving concentrate quality and resulting in a positive economic impact.

Practical Justification

The identification of species is essential for characterizing each mineralogical formation involved in the concentration process. This understanding is important to optimize the recovery rate of the concentrate through flotation of polymetallic ores, considering reagent dosage and pulp alkalinity variables, in the Andean regions of Peru. Furthermore, the findings of this study can serve as a model for other mines with similar mineralogy, improving lead and silver recovery using experimental designs in flotation.

Economic Justification

Effective management of raw materials is essential for optimizing the production of metallurgical mining resources, which can significantly enhance the revenue of a concentrator plant.

Social Justification

From a social perspective, optimizing resource use in the processing of polymetallic ores through the flotation process benefits both directly and indirectly involved communities. Thus, this approach helps guarantee local employment and fosters community development for future generations.

METHODOLOGY

This research employs a quantitative, deductive, and experimental design methodology. It applies knowledge of mineralogical characterization and analyzes the variables influencing the flotation process, which facilitates the optimization of the lead-silver concentrate recovery rate. The research follows a specific design framework:

Research Method Procedure

- **Sampling and Sample Preparation**

  A representative sample of polymetallic ore was collected from the Esmeralda, Orión, and Hércules adits. Using a jaw crusher, this sample was then ground to 100% passing through a 10 mesh. To ensure thorough research testing, the entire sampled batch was quartered, and a portion was stored as a control sample.

- **Chemical Assays and Mineralogical Characterization**

  A representative ore sample was ground in a ball mill to determine the head grades of lead (Pb) and silver (Ag). Additionally, samples of different gradation (+100 mesh, <-100, +200] mesh, and <-200, +400] mesh) from the study adits were sent to the mineralogical characterization laboratory to identify the mineral species present, assess volumetric distribution, determine degrees of liberation, and evaluate associations or bonds present. The mineralogical analysis was performed using a polarized light microscope.

Figure 1. Methodological scheme of the research.

Note. The figure illustrates the measurements included in the research design for this study.

Source: Prepared by the authors.

O_1: Mineralogical characterization of the Esmeralda, Orión, and Hércules adits.

X: Development of the Plackett-Burman experimental design and analysis of the variables influencing the metallurgical flotation process.

O_2: Optimization of lead and silver recovery in the high Andean region of Peru.

- **Experimental Designs Using Minitab 19**

  Plackett-Burman experimental designs were generated using Minitab 19 statistical software to assess the independent variables that significantly influence the recovery and quality of lead-silver concentrate, as well as to optimize the flotation process. A test plan was established based on the Plackett-Burman template provided by the software to assess the impact of the reagent blend (AP-3418/AR-404, AR-242) and the pH of the pulp on the recovery and quality of the lead-silver concentrate. Other process variables were kept constant during the laboratory metallurgical flotation tests.

RESULTS

Chemical Assays

The chemical assays for lead (Pb) in the study adits were conducted using the volumetric analysis, while silver (Ag) head grades were determined through cupellation. Laboratory reports indicated that the Esmeralda adit exhibited the highest silver head grade (6.65 oz/t), whereas the Hércules adit showed the lowest silver head grade (1.12 oz/t). Additionally, the reports on lead concentration revealed that the Orión adit had the highest lead head grade (1.46%). The results of the chemical assays conducted in the study adits are summarized in Table 1 below.

Table 1. *Chemical Assays Performed on Samples from the Adits.*

Adit	Ag head grade (oz/t)	Pb head grade (%)
Esmeralda	6.65	1.22
Orión	1.59	1.46
Hércules	1.12	0.89

Note. The table displays the Ag-Pb head grades for the Esmeralda, Orión, and Hércules adits.

Source: Prepared by the authors.

Volumetric Distribution of the Esmeralda, Orión, and Hércules Adits.

The analysis of a representative sample from the Esmeralda adit, observed under a polarizing light microscope, revealed that the primary lead-bearing minerals are galena and lead sulfosalts. These minerals were predominantly found in the particle size range of (-74 µ + 37 µ). Contaminating minerals such as arsenopyrite were identified, with a significant distribution of 3.62%. Sphalerite I and II were also identified, showing distribution values of 2.4% and 0.48%, respectively. The presence of fahlores was noted with a distribution of 0.19%, and stibnite was found with a distribution of 0.33%, all achieved at the particle size distribution range of (-74 µ + 37 µ).

The mineralogical characterization of a representative sample from the Orión adit determined that galena is the main lead-bearing species. The highest distribution of this species occurs at a particle size smaller than 74 µ, with a reported value of 1.02%. Contaminant ores, such as arsenopyrite, were also identified, showing the highest distribution of 3.81% at a particle size smaller than 200 mesh. Sphalerite I and II were also identified, showing distribution values of 2.61% and 0.35%, respectively, at a particle size smaller than 200 mesh. The presence of fahlores was noted with a maximum value of 0.08% retained on 200 mesh, and stibnite was found with a distribution of 0.41% achieved at a particle size distribution range of 100% -200 mesh.

The analysis conducted for the Hércules adit revealed that galena is the primary lead-bearing mineral, with a maximum distribution value of 2.21% at a particle size smaller than 200 mesh. Arsenopyrite was also detected with a maximum distribution of 4.95% at particles larger than 74 µ, while type I sphalerite exhibited a maximum distribution of 13.25% at particles smaller than 74 µ. Notably, the volumetric distribution of type I sphalerite was the highest compared to other adits. It is important to mention that fahlores, type II sphalerite, and stibnite were not reported.

The volumetric distribution values of all minerals identified under the reflected light optical microscope are shown in Table 2, categorized by gradation: +100 mesh, <-100, +200] mesh, and <-200, +400] mesh. The data shows that these minerals are found in greater volumes at finer particle sizes, suggesting that the species are liberated in this range of particle size distribution.

Degree of Liberation of Minerals from Esmeralda, Orión, and Hércules Adits

The mineral liberation analysis conducted at Esmeralda adit to determine the degree of liberation of galena and sulfosalts reported values of 86.08% and 78.82%, respectively, at a particle size of 100% -200 mesh. Regarding contaminants, arsenopyrite exhibited a degree of liberation of 95.06%; fahlores showed a degree of liberation of 76.88%; sphalerite revealed a degree of liberation of 100%; and stibnite showed a degree of liberation of 91.66%. These values were determined at a particle size smaller than 200 mesh. Additionally, the highest degree of liberation for type I sphalerite was found at a particle size of 100% -100 mesh.

The mineral liberation analysis conducted at Orión adit revealed that galena exhibited a degree of liberation value of 91.72% at a particle size smaller than 200 mesh. At this same particle size distribution, the maximum degrees of liberation were found for type I sphalerite, arsenopyrite, and stibnite, with values of 95.2%, 95.11%, and 97.63%, respectively. Notably, type II sphalerite exhibited the highest degree of liberation of 93.55% for material retained on 100 mesh, while fahlores showed a value of 88.9% for material retained on 200 mesh.

The mineral liberation analysis conducted at Hércules adit revealed that galena was found to be 97.62% liberated at a particle size of 100% -200 mesh. In contrast, the minerals considered detrimental to the process such as sphalerite I, sphalerite II, and arsenopyrite reported liberation values of 96.14%, 44.89%, and 97.38%, respectively, at a particle size range of <-100, +200] mesh. The total degrees of liberation for the identified species at various gradations are summarized in Table 3.

Types of bonding between Pb and Ag

A representative sample from the Esmeralda adit was analyzed to determine the bonds present. Considering a particle size of 100% +100 mesh, galena was found to form bonds with pyrite, gangue, fahlores, and type I sphalerite, all of which were reported to be moderately easy to liberate. At a particle size of 100% -100mesh, galena formed bonds with arsenopyrite, pyrite, gangue, and fahlores. The bonds formed with gangue, pyrite, and arsenopyrite were reported to be very difficult to liberate. At a particle size of 100% -200 mesh, galena formed bonds with fahlores, lead sulfosalts, type I sphalerite, arsenopyrite, stibnite, and chalcopyrite. Among these, the bonds with fahlores were described as moderately difficult to liberate, while those with lead sulfosalts were classified as very difficult.

Similarly, a representative sample from the Orión adit was also analyzed to determine the bonds present. Considering a particle size of 100% +100 mesh, galena formed bonds with arsenopyrite, which was found to be very difficult to liberate. At a particle size of 100% -100 mesh, the bonds formed between galena and chalcopyrite were deemed very difficult to liberate. Finally, at a particle size of 100% -200 mesh, the bond formed between galena and arsenopyrite was considered impossible to liberate.

In contrast, the bonds formed between galena and other minerals found in a representative sample from the Hércules adit were reported to be easy to liberate.

A comprehensive description of the observed bonds and their liberation potential for the various samples is detailed in Table 4. Additionally, Figures 2, 3, and 4 depict the main micrographs of galena from the study adits.

Plackett-Burman experimental design for the Esmeralda, Orión, and Hércules adits

Flotation tests were conducted on representative samples from the three mine adits under study, focusing on the effects of the reagent blend (AP-3418/AR-404), AR-242, and the pH of the pulp. The tests evaluated the following parameters: reagent blend concentrations of 18, 24, and 30 g/t; AR-242 concentrations of 5, 10, and 15 g/t; and pH values of 10.5, 11.0, and 11.5. In the Esmeralda adit, the highest lead (Pb) recovery was 84.60%, which was achieved during run order 3. The best grade concentrate achieved was 60.24%, corresponding to run order 6. Additionally, the highest silver (Ag) recovery was 84.96%, with a head grade of 341.58 oz/t, attained in run order 4. The results of the total tests for the experimental design of the Esmeralda adit are detailed in Table 5.

Table 2. *Volumetric Distribution of Mineralogical Species from the Esmeralda, Orión, and Hércules Adits.*

PIT 1: VOLUMETRIC DISTRIBUTION OF MINERALOGICAL SPECIES FROM THE ESMERALDA ADIT															
MESH	GAN-GUES	PYRITE	ARSE-NOPYRITE	GALENA	CHALCO-PYRITE	FAHLO-RE	SPHALERI-TE I	SPHALE-RITE II	MAGNE-TITE	STIBNITE	MARCA-SITE	Pb SUL-FOSALTS	PIRRO-TYTE	HEMA-TITE	RUTILE
+100	91.19	4.07	0.97	0.17	0	0	0.28	-	0	0.02	-	-	-	-	-
<-100, +200]	87.81	6.05	1.7	0.3	-	0	1.78	0.45	0.04	0.13	-	-	-	-	-
<-200, +400]	82.25	7.43	3.62	0.88	0.1	0.19	2.4	0.48	0.1	0.33	0	0.1	0.1	-	-
PIT 2: VOLUMETRIC DISTRIBUTION OF MINERALOGICAL SPECIES FROM THE ORIÓN ADIT															
(+100)	86.9	5.88	1.02	0.28	0	-	0.46	0.28	0	0	0	-	-	0	-
<-100, +200]	84.15	6.41	2.18	0.81	0	0.08	1.94	0.32	0.04	0.24	0.2	-	-	0	0.04
<-200, +400]	82.08	6.97	3.81	1.02	0.12	0.06	2.61	0.35		0.41	0.12	-	-	-	0.12
PIT 3: VOLUMETRIC DISTRIBUTION OF MINERALOGICAL SPECIES FROM THE HÉRCULES ADIT															
(+100)	61.49	17.03	1.41	0.69	0	-	0.86	-	-	-	0	-	0.36	-	-
<-100, +200]	63.59	21.06	4.95	1.25	0.18	-	3.54	0	-	-	0.75	-	0.25	-	0.23
<-200, +400]	58.85	15.13	4.06	2.21	0.19	-	13.25	-	-	-	0.17	-	1.52	-	-

Note. The table above shows the volumetric distribution for mesh sizes +100, <-100, +200], and <-200, +400].
Source: Prepared by the authors.

Table 3. *Degrees of Liberation of Mineralogical Species from the Esmeralda, Orión, and Hércules Adits.*

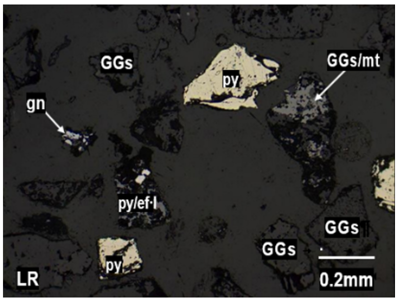
PIT 1: DEGREE OF LIBERATION OF MINERALOGICAL SPECIES FROM THE ESMERALDA ADIT															
MESH	GAN-GUES	PYRITE	ARSE-NOPYRITE	GALENA	CHALCO-PYRITE	FAHLO-RE	SPHALERI-TE I	SPHALE-RITE II	MAGNE-TITE	STIBNITE	MARCA-SITE	Pb SUL-FOSALTS	PIRRO-TYTE	HEMA-TITE	RUTILE
+100	99.07	91.4	78.91	69.6	0.24	3	68.22	-	3.5	42.57	-	-	-	-	-
<-100, +200]	99.72	97.2	89.32	78.08	-	65.25	95.39	93.92	63.55	87.28	-	-	-	-	-
<-200, +400]	99.99	98.1	95.06	86.08	67.22	76.88	94.23	100	100	91.66	72.25	78.82	90.59	-	-
PIT 2: DEGREE OF LIBERATION OF MINERALOGICAL SPECIES FROM THE ORIÓN ADIT															
(+100)	98.78	94	71.19	71	9	-	60.94	93.55	0	29.09	7.92	-	-	0	-
<-100, +200]	99.23	96.3	87.79	86.14	2.62	88.9	87.51	87.18	45.69	76.49	76.33	-	-	1	58.18
<-200, +400]	99.77	96.9	95.11	91.72	70.54	75.3	95.2	88.79	-	97.63	74.59	-	-	-	74.59
PIT 3: DEGREE OF LIBERATION OF MINERALOGICAL SPECIES FROM THE HÉRCULES ADIT															
(+100)	96.14	92.1	63.78	54.71	60.93	-	56.37	-	-	-	28.75	-	69.34	-	-
<-100, +200]	98.73	98.4	97.38	93.11	94.85	-	96.14	44.89	-	-	73.32	-	86.35	-	100
<-200, +400]	98.39	97.2	91.1	97.62	70.89	-	95.21	-	-	-	100	-	96.09	-	-

Note. The table above shows the degrees of liberation for mesh sizes +100, <-100, +200], and <-200, +400].
Source: Prepared by the authors.

Table 4. *Types Of Bonding at Various Particle Size Distribution Ranges for the Esmeralda, Orión, and Hércules Adits.*

Particle size	Esmeralda Adit			Orión Adit			Hércules Adit		
	Bonding	Vol. (%)	Liberation potential	Bonding	Vol. (%)	Liberation potential	Bonding	Vol. (%)	Liberation potential
+100 mesh	py/gn GGs/gn GGs/py/gn GGs/gn/Fhls GGs/py/gn/sph I	0.04 0.08 0.25 0.08 0.08	Moderately easy Moderately easy Moderately difficult Moderately easy Moderately easy	gn/sph I GGs/gn apy/gn py/gn/sph I GGs/apy/gn GGs/gn/sph I	0.09 0.23 0.19 0.09 0.09 0.19	Easy Very difficult Very difficult Moderately easy Moderately easy Moderately easy	py/gn gn/sph I	1.79 0.83	Easy Moderately easy
<-100, +200] mesh	apy/gn py/gn GGs/gn gn/Fhls GGs/py/gn py/apy/gn	0.08 0.08 0.08 2.00 0.08 1.00	Easy Easy Very difficult to impossible Moderately easy Moderately difficult Very difficult	gn/sph I apy/gn cp/gn GGs/gn GGs/cp/CGRs	0.24 0.08 0.08 0.16 0.08	Easy Easy Very difficult to impossible Moderately easy Moderately easy	gn/sph I, gn/sph II	0.02 0.16	Easy Easy
<-200, +400] mesh]	gn/Fhls gn/Pb sulfosalts gn/sph I apy/gn gn/stb cp/gn/CGRs py/gnCGRs	0.10 0.29 0.10 0.19 0.10 0.10 0.10	Moderately difficult Very difficult Easy Moderately easy Easy Moderately easy Moderately easy	gn/sph I py/gn apy/gn	0.12 0.12 0.23	Moderately easy Moderately easy Impossible	sph I/gn GGs/apy/gn	0.11 0.17	Moderately easy Moderately easy

Note. The table shows the galena forming bonds with other ores and their liberation potential.
Source: Prepared by the authors.

<table>
<tr><th colspan="3">MAIN MICROGRAPHS OF GALENA IN THE STUDY ADITS</th></tr>
<tr><th>ESMERALDA</th><th>ORIÓN</th><th>HÉRCULES</th></tr>
<tr>
<td>
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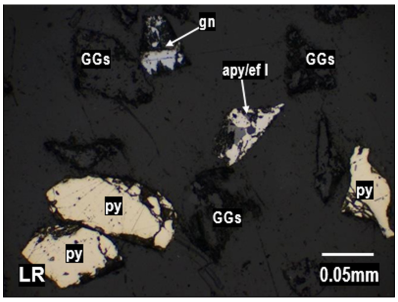
<td>
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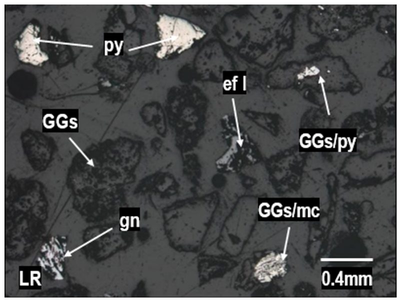
<td>
</td>
</tr>
<tr>
<td>Figure 2 Liberated grains of gangues (GGs), pyrite (py), galena (gn), mixed gangue/magnetite (GGs/mt), and pyrite/sphalerite I (py/sphI) found in particle sizes of +100 mesh.</td>
<td>Figure 3 Liberated grains of gangues (GGs), pyrite (py), galena (gn), and arsenopyrite/sphalerite I (apy/sph I) found in the particle size distribution range of <-100, +200] mesh.</td>
<td>Figure 4 Liberated grains of pyrite (py), sphalerite (sph I), galena (gn) and gangues (GGs), binary mixed particles of gangues with marcasite (GGs/mc) found in particle sizes of +100 mesh.</td>
</tr>
</table>

Note. The figures show the liberated and interlocked grains in the study adits.

Source: Prepared by the authors.

In the Orión adit, the maximum lead (Pb) recovery was 83.73%, achieved in run order 2, while the highest grade of lead (Pb) concentrate, 61.63%, was obtained from run order 7. The highest silver (Ag) recovery was 82.50%, achieved in run order 4, while the best silver (Ag) head grade of 85.35 oz/t was achieved in run order 3. The experimental design results for the Orión adit are provided in Table 6.

In the Hércules adit, the best lead (Pb) recovery was 83.79%, as recorded in run order 9. The highest grade of lead (Pb) concentrate, 56.25%, was achieved in run order 2. The highest silver (Ag) recovery recorded was 82.57%, obtained in run order 3, while the best head grade was 63.66 oz/t, noted in run order 6. The total experimental design tests are summarized in Table 7.

Standardized effects analysis

Table 8 outlines the influence of the variables pH, reagent blend (AP-3418/AR-404), AR-242, and their interactions on the recovery and concentrate quality responses for the study adits.

Optimization of flotation parameters for polymetallic ore

The independent variables that most significantly contributed to the flotation process of the three adits under study were optimized using Minitab 19. For the Esmeralda adit, the optimization results indicated a dosage of 18 g/t of reagent blend, 5 g/t of Ar-242, and a pH of 11.5. For the Orión adit, the optimization results indicated a dosage of 30 g/t of reagent blend, 5 g/t of Ar-242, and a pH of 10.5. Lastly, for the Hércules adit, the optimization results indicated a dosage of 30 g/t of reagent blend, 15 g/t of Ar-242, and a pH of 11.5. The results and optimization plots for the flotation of minerals from the Esmeralda, Orión, and Hércules adits can be found in Figures 5, 6, and 7.

HYPOTHESIS TESTING

The results of the Plackett-Burman experimental design for the Esmeralda adit, which was developed based on the mineralogical characterization of the adit, are shown in Table 5. Run order 2 identifies the optimal levels of recovery and grade of lead-silver concentrate, determined by the dosage of reagents. The results of test 2 differ from the other runs for this adit in terms of test conditions, thereby confirming the hypothesis.

For the Orion adit, the experimental design was similarly based on its mineralogical characterization. The findings indicate that run order 3 achieves the optimal values for the recovery and grade of the lead-silver concentrate, which are influenced by the independent variable of reagent dosage. This result significantly differs from those of the other tests, as illustrated in Table 6, thus confirming the hypothesis.

Table 5. *Plackett-Burman Experimental Design and Results of the Metallurgical Tests Conducted at the Esmeralda Adit.*

Run order	pH	Reagent blend (AP-3418/AR-404)	AR-242	pH	Reagent Blend (AP-3418/AR-404)	AR-242	Pb recovery (%)	Pb head grade (%)	Ag recovery (%)	Ag head grade (oz/t)
1	1	-1	-1	11.5	30	5	83.68	57.24	84.72	297.54
2	1	-1	-1	11.5	18	5	82.00	58.66	83.79	310.64
3	1	1	1	11.5	30	15	84.60	56.25	84.91	326.46
4	0	0	0	11	24	10	83.30	56.27	84.96	341.58
5	0	0	0	11	24	10	83.01	56.62	84.80	339.41
6	-1	-1	-1	10.5	18	5	81.20	60.24	82.15	312.07
7	-1	1	1	10.5	30	15	83.24	55.46	83.47	310.58
8	-1	1	-1	10.5	30	5	82.21	59.25	81.23	295.10
9	-1	-1	1	10.5	18	15	82.36	57.78	82.06	290.00
10	1	-1	1	11.5	18	15	80.77	56.80	80.82	335.07

Note. The table presents the operational variables used for the experimental design.
Source: Prepared by the authors.

Table 6. *Plackett-Burman Experimental Design and Results of the Metallurgical Tests Conducted at the Orión Adit.*

Run order	pH	Reagent blend (AP-3418/AR-404)	AR-242	pH	Reagent Blend (AP-3418/AR-404)	AR-242	Pb recovery (%)	Pb head grade (%)	Ag recovery (%)	Ag head grade (oz/t)
1	0	0	0	11	24	10	83.18	57.65	81.58	74.58
2	0	0	0	11	24	10	83.73	57.15	81.85	73.22
3	-1	1	-1	10.5	30	5	81.89	61.39	81.44	85.35
4	-1	1	1	10.5	30	15	82.58	59.94	82.50	84.84
5	1	1	-1	11.5	30	5	82.24	58.93	81.17	77.67
6	-1	-1	-1	10.5	18	5	82.32	61.60	79.44	78.24
7	1	-1	1	11.5	18	15	78.34	61.63	75.68	76.22
8	-1	-1	1	10.5	18	15	83.71	59.30	81.31	77.55
9	1	1	1	11.5	30	15	82.28	59.34	79.10	79.34
10	1	-1	-1	11.5	18	5	78.97	60.69	78.83	76.68

Note. The table presents the operational variables used for the experimental design.
Source: Prepared by the authors.

Table 7. *Plackett-Burman Experimental Design and Results of the Metallurgical Tests Conducted at the Hércules Adit.*

Run order	pH	Reagent blend (AP-3418/AR-404)	AR-242	pH	Reagent Blend (AP-3418/AR-404)	AR-242	Pb recovery (%)	Pb head grade (%)	Ag recovery (%)	Ag head grade (oz/t)
1	0	0	0	11	24	10	83.30	51.94	78.26	38.30
2	1	-1	-1	11.5	18	5	82.53	56.25	79.38	36.94
3	-1	-1	1	10.5	18	15	83.29	48.11	82.57	43.38
4	-1	1	-1	10.5	30	5	82.83	49.29	82.16	57.07
5	0	0	0	11	24	10	83.40	52.26	78.36	36.88
6	1	1	-1	11.5	30	5	80.18	56.16	78.29	63.66
7	-1	-1	-1	10.5	18	5	81.31	47.17	81.82	41.29
8	1	1	1	11.5	30	15	82.40	54.12	80.88	62.63
9	-1	1	1	10.5	30	15	83.79	48.13	80.94	55.69
10	1	-1	1	11.5	18	15	84.54	54.67	80.92	37.94

Note. The table presents the operational variables used for the experimental design.
Source: Prepared by the author.

Table 8. *Summary of the Interactions of the Main Variables.*

Adit	Response	Variables
Esmeralda	Lead (Pb) head grade	AR-242
	Lead (Pb) recovery	AP-3418/AR-404
	Silver (Ag) head grade	pH, (pH – AR-242) interaction
	Silver (Ag) recovery	AP-3418/AR-404, pH, (AP-3418/AR-404 – AR-242) interaction, (pH – AR-242) interaction, (pH – AP-3418/AR-404) interaction
Orión	Lead (Pb) head grade	Interaction (pH – AR-242), (pH – AP-3418/AR-404) interaction, AP-3418/AR-404
	Lead (Pb) recovery	Interaction (pH – AR-242), AR-242, AP-3418/AR-404
	Silver (Ag) head grade	AP-3418/AR-404, A, (pH – AP-3418/AR-404) interaction
	Silver (Ag) recovery	pH, AP-3418/AR-404, (pH – AR-242) interaction
Hércules	Lead (Pb) head grade	pH
	Lead (Pb) recovery	AR-242, interaction (pH – AP-3418/AR-404)
	Silver (Ag) head grade	AP-3418/AR-404
	Silver (Ag) recovery	pH, AP-3418/AR-404, (pH – AR-242) interaction, (pH – AP-3418/AR-404 – AR-242) interaction

Note. The table depicts the interactions observed in the Esmeralda, Orión, and Hércules adits.

Source: prepared by the authors.

Similarly, for the Hercules adit, the development of the experimental design and selection of reagents for the flotation tests were also based on its mineralogical characterization. The results from this series of tests demonstrate that run order 8 yields the optimal values for recovery and grade of the lead-silver concentrate, as shown in Table 7. These findings differ significantly from the other runs, further supporting the hypothesis.

DISCUSSION

The results obtained in this research allowed for the optimization of lead-silver (Pb-Ag) recovery rate by flotation through mineralogical and metallurgical characterization, as well as the optimal design of the flotation process of polymetallic ore. The mineralogical characterization revealed that most valuable particles exhibited a simple intergrowth texture, which proved beneficial for the concentration process. On the other hand, the experimental design showed that to enhance metallurgical performance for recovering lead and silver from the Esmeralda adit, 18 g/t of reagent blend (AP-3418 and AR-404), 5 g/t of AR-242, and an optimal pH of 11.50 were necessary. For the Orion adit, the optimal parameters included 30 g/t reagent blend (AP-3418 and AR-404), 5 g/t of AR-242, and a pH of 10.50. Similarly, for the Hercules adit, the optimal parameters were 30 g/t of reagent blend (AP-3418 and AR-404), 15 g/t of AR-242, and a pH of 11.50.

In comparing the work of Ramos and Orihuela (2017) with this study, which aimed to evaluate complex polymetallic minerals through flotation metallurgical tests to obtain copper, lead, and zinc concentrates with satisfactory recoveries and grades that meet smelting requirements, it was concluded that the best conditions for copper-lead separation involved a dosage of activated carbon of 233.3 g/t, 60 g/t of reagent blend BCS, 10 g/t of collector Ap 5100, and maintaining the pH under natural conditions for 7 minutes. This resulted in a copper concentrate grade of 25.6% and a lead concentrate grade of 54.3%. In contrast, this research determined that optimal metallurgical performance in recovering lead and silver required a reagent blend dosage ranging from 18 to 30 g/t (AP-3418 and AR-404), AR-242 dosages ranging from 5 to 15 g/t, and a pH level ranging from 10.5 and 11.5, as detailed in Tables 5, 6, and 7.

CONCLUSIONS

The mineralogical characterization of representative samples from the study adits identified several minerals. In adit 1, the identified minerals were pyrite, arsenopyrite, galena, chalcopyrite, fahlores, sphalerite I, magnetite, stibnite, sphalerite II, marcasite, lead sulfosalts, and pyrrhotite. In adit 2, the minerals found were pyrite, arsenopyrite, marcasite, galena, chalcopyrite, sphalerite I, sphalerite II, magnetite, stibnite, hematite, rutile, and fahlores. Finally, in adit 3, the minerals found were pyrite, arsenopyrite, marcasite, galena, chalcopyrite, pyrrhotite, sphalerite I, sphalerite II, and rutile. Most of the minerals of interest exhibited a simple intergrowth texture, while a smaller fraction showed a more complex intergrowth structure. This

ESMERALDA ADIT

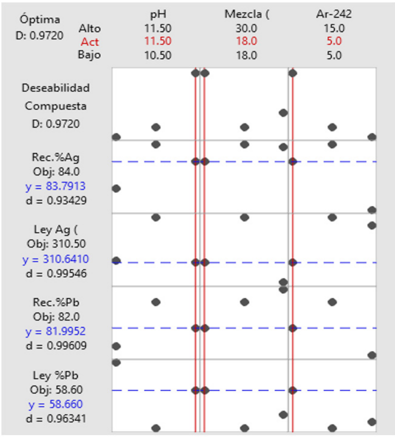


Figura 5. Optimization plot of the flotation parameters for pit 01.

ORIÓN ADIT

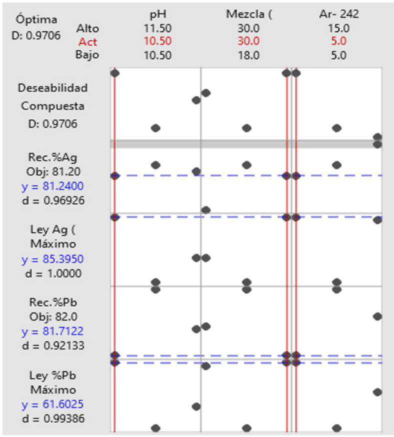


Figura 6. Optimization plot of the flotation parameters for pit 02.

HÉRCULES ADIT

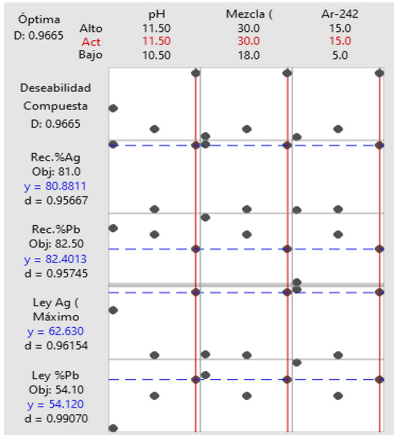


Figura 7. Optimization plot of the flotation parameters for pit 03.

Note. The table presents the optimization plot of the independent and dependent variables in the three study adits.
Source: Prepared by the authors.

complexity can enhance the separation, flotation, and concentration processes of the minerals of interest.

The standardized analysis of effects in the experimental design allowed for identifying the interactions of variables (A: pH, B: reagent blend, C: collector AR-242) that significantly influence the head grade and recovery rate of lead (Pb) and silver (Ag). Consequently, the parameters for optimizing metallurgical performance were established as follows: For adit 1, the recommended dosage is 18 g/t of reagent blend (AP-3418 and AR-404), 5.0 g/t of a secondary collector AR-242, and a pH of 11.50. For adit 2, the dosage is 30 g/t of reagent blend (AP-3418 and AR-404), 5.0 g/t of secondary collector AR-242, and a pH of 10.50. For adit 3, the dosage is 30 g/t of reagent blend (AP-3418 and AR-404), 15.0 g/t of secondary collector AR-242, and a pH of 11.50.

Operational variables such as particle size, air flow in flotation cells, and conditioning times contribute to establishing a recovery model and determining the quality of concentrates from a geometallurgical perspective.

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Rosa María Tiburcio Alva (co-author): Formal analysis, methodology, supervision, and validation.

Sharon Elisa Aguilar Zevallos: Investigation and writing (review & editing).