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## Froth flotation of Aljustrel sulphide complex ore

Rui Sousa<sup>\*</sup>, Aurora Futuro<sup>\*</sup>, Carlos Setas Pires<sup>\*\*</sup>, Mário Machado Leite<sup>\*\*\*</sup>

<sup>\*</sup> Departamento de Engenharia de Minas, Faculdade de Engenharia, Universidade do Porto, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal. Corresponding author: rui.sousa@lneg.pt (Rui Sousa)

<sup>\*\*</sup> Almina, Minas do Alentejo SA, Algaes, 7600-015 Aljustrel, Portugal

<sup>\*\*\*</sup> Laboratório Nacional de Energia e Geologia, Rua da Amieira, Apartado 1089, 4466-901 S. Mamede de Infesta

**Abstract:** Froth flotation of copper-zinc-lead ores is a complex process based on similar metallurgy processing and strong interactions between chalcopyrite and sphalerite. These types of ores represent 15% of the world production and 7.5% of copper deposits all over the world. In the present study, an attempt is made to assess the feasibility of froth flotation of a complex sulphide ore, where the effect of liberation grade as well as depressant, collector and frother dosage was studied. Copper flotation is dependent on the mineral liberation grade, which is significantly related with the size distribution. It was shown that longer grinding time increased the Cu grade but decreased the copper recovery due to the presence of very fine particles. Lime and sodium metabisulphite, used as depressants, allowed to achieve a good flotation performance. It was shown that for this type of ore lower dosage of the collector should be applied. Finally, a higher frother dosage led to a greater selectivity and reduced entrainment of very fine particles, but also caused low kinetic ratio and low recovery of valuable minerals. A well-defined flotation objective was imperative for a good liberation grade and reagents dosage selection, mainly for complex sulphide ores. A useful contribute was given to a better technical understanding of flotation of a complex sulphide ore from the Moinho deposit.

**Keywords:** *froth flotation, mineral liberation, flotation reagents, complex sulphide ore*

## Introduction

Flotation can be defined as a physical-chemical process that allows for separation based on differences between surface properties of valuable and unwanted gangue minerals. Flotation comprises three mechanisms, that is *i*) adsorption of specific reagents on the surface of valuable minerals and their attachment to air bubbles; *ii*) entrainment of hydrophilic particles; and *iii*) physical entrapment between particles in the froth attached to air bubbles (aggregation). Controlling the stability of froth phase is important to achieve adequate separation (Wills and Napier-Munn, 2006; Kowalczyk and Drzymala, 2017).

Froth flotation occurs in an aqueous medium which involves aggregation of air bubbles and mineral particles with subsequent levitation of aggregates to the surface and transfer to a froth phase. The degree of particle surface wettability determines attachment and aggregation. When a solid surface does not have affinity to water, the surface is said to be hydrophobic and an air bubble attaches to the surface being floated (Fuerstenau et al., 1985). Froth flotation has become the most important method of mineral processing, which is pointed out by the economic relevance of the chemical industry (Fuerstenau and Urbina, 1987). A flotation system includes several interrelated components, that is chemical compounds (collectors, frothers, activators, depressants and pH regulators), equipment (cell design, agitation, air flow, cell bank configuration and control) and operational parameters (feed rate, mineralogy, particle size, pulp density and temperature) (Klimpel., 1995).

It is possible, in froth flotation, to realize that the particle size distribution influences both the physics of a process by controlling the collision mechanism between particles and bubbles, and the degree of liberation (Machado Leite, 1992). The reagent type and dosage also has a strong effect on the flotation behaviour, and should be carefully analyzed in order to improve the recovery of useful components. The effective usage of reagents that allows for maximizing metallurgical results and minimizing operating costs is one of the main concerns for researchers and operators (Greet et al., 2010). Frothers have an important effect on flotation performance (Barbian et al., 2005; Bulatovic, 2007; Wang et al., 2016), reducing the bubble size, preventing coalescence and stabilizing the froth (Cho and Laskowski, 2002). An optimized frother behaviour could reduce the entrainment of very fine particles, which play a critical role in processing of ores with a large proportion of fine gangue particles (Yianatos et al., 2015; Wang et al., 2016).

Making a decision on laboratory tests of mineral processing is not a straightforward procedure. Results obtained from batch flotation tests could be the basis for decision and plans for further experiments (Kostovic and Gligoric, 2015). Therefore, this work pretends to extend the available alternatives for the study of sulphide ore from the Moinho deposit from the Aljustrel Mine. In order to that, the effect of the collector, depressant and frother dosages (chemistry components) and the degree of mineral liberation related with the particle size (operational component) were tested. The recovery and grade are commonly used to describe the efficiency of metallurgical processes (Fuerstenau and Han, 2011), therefore the results were graphically represented by the Mayer upgrading plot to evaluate and compare the copper flotation performance.

## **Materials and methods**

### **Material**

Complex sulphide ore samples were obtained from the Moinho deposit (Aljustrel mine), which is a volcanic massive sulphide complex ore found in southern Portugal.

The materials were collected after the first fragmentation step of Aljustrel plant, and then crushed, homogenized and divided into 1 kg bags. The ore consisted in a complex association of chalcopyrite, galena and sphalerite, with pyrite, quartz and carbonates being the major gangue minerals. A chemical analysis was performed and the following composition was determined: 1.61% of Cu, 1.54% of Zn, 0.55% of Pb, 35.19% of Fe, and others. The size distribution analysis showed  $K_{80}=1.51$  mm for the crushed material (Fig. 1).

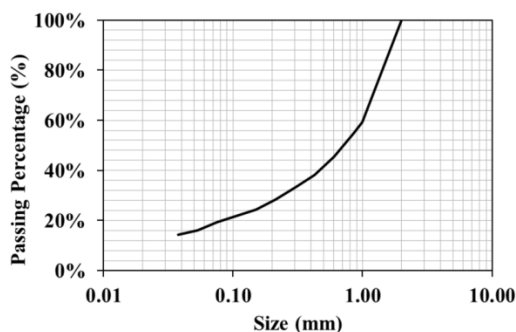


Fig. 1. Particle size distribution of an ore sample

### Chemical reagents

All the chemical reagents used in this study were supplied by Almina, Minas do Alentejo SA. Calcium oxide (CaO) was used as a pH regulator and a depressant for pyrite minerals to avoid the sphalerite pre-activation. Sodium metabisulfite (MBS) was used as a depressant for pyrite and sphalerite, due to the capacity to avoid collector adsorption and elimination of hydrophobic species on the mineral surface (Pattison, 1981; Shuhua, 2006). Dialkyl dithiophosphate and thionocarbamate were used as collectors, and methyl isobutyl carbinol (MIBC) as a frother.

### Batch flotation and grinding tests

Grinding tests were applied in order to achieve an adequate degree of mineral liberation. During grinding, addition of a depressant was performed to reduce flotation of unwanted minerals. The grinding tests were performed in a grinding mill of 8250 cm<sup>3</sup>. A total weight of 7 kg of steel bars was used as grinding media for 1 kg of ore with water at 60% of the weight of solids. The depressant was added to grinding. Next, the material was transferred to a 2330 cm<sup>3</sup> flotation cell along with the necessary water in order to obtain a pulp density of 40%. Agitation was kept at 2150 rpm for the impeller speed, and aeration lasted five minutes. Aeration was needed to depress pyrite (Konisgmann, 1973; Ek, 1985) and to improve selectivity between sulphide minerals, contributing to better metallurgical results (Senior and Traliar, 1991; Silva, 1999; Asian et al., 2003). Following this, pH was adjusted at 10.5 with CaO. Next, the

chalcopyrite promoter was added and two minutes later the frother was added. One minute later, the air valve was opened and flotation started with the first concentrate collection lasted three minutes. Next, the air valve was closed, pH adjusted and the promoter added. The air valve was opened again, and the second concentrate was collected during two minutes. New pH regulation and promoter addition were carried out prior the third flotation period to recover a concentrate during six minutes. These three concentrates ( $t = 3, 5$  and  $11$  min) were dried, weighed and analysed to calculate the recovery of Cu, Fe, Zn and Pb. Using this data, it was possible to draw the Mayer upgrading curve for all performed tests.

## Results and discussion

### Effect of liberation grade

The mineral liberation grade depends on size reduction. Extending the grinding time should increase the liberation degree by increasing the free surface area. Particles with size larger than  $0.30$  mm and smaller than  $0.01$  mm are difficult to float. Since very fine particles have a tendency for entrainment, the biggest challenge for grinding was to achieve the best particle size distribution with the best liberation grade, while simultaneously minimizing production of fine particles. In order to do that, three different grinding times, that is  $30, 60$  and  $90$  minutes, were applied. Then, batch flotation tests were carried out under the same operational conditions: pH =  $10.5$ , agitation speed of  $2150$  rpm, CaO =  $2500$  g/Mg, promoter =  $30$  g/Mg, frother =  $7$  g/Mg.

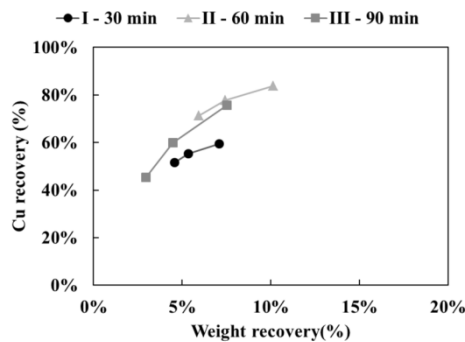


Fig. 2. Effect of grinding time on copper and mass (concentrate yield) recovery (Test I – 30 min, Test II – 60 min and Test III – 90 min)

The balance between copper and mass recovery (namely concentrate yield) is illustrated in Fig. 2 as the Mayer diagram (upgrading plot). Table 1 shows the final (cumulated at the end of 11 minutes) recovery of main elements, and concentrate grade.

Table 1. Recovery and grade of main elements  
(Test I – 30 min, Test II – 60 min and Test III – 90 min)

Test	Cu		Zn		Pb		Fe	
	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %
I	14.78	59.39	2.68	12.81	2.73	33.63	33.58	6.43
II	12.28	83.69	2.48	16.96	2.66	48.38	35.23	9.55
III	15.16	75.56	1.51	7.55	1.73	23.49	34.68	7.03

Shorter grinding time (30 minutes) resulted in a low Cu recovery and higher Cu grade. This indicated on the insufficient grinding time, which was not enough to liberate copper minerals and produced a significant amount of particles too coarse to be floated. Flotation performed after 60 min (Test II) of grinding showed the best performance, as seen in the Mayer plot (Fig. 2), presenting a better balance between metal recovery and concentrate yield (weight recovery). Test III resulted in a better selectivity, with lower recovery of unwanted elements (Zn, Pb and Fe). Increasing the grinding time allowed to decrease the unwanted minerals in the concentrate and increase the copper grade. This fact could be justified by the higher liberation grade, which promoted the increased efficiency of chemical reagents. Decrease in the copper recovery with increased grinding time (60 to 90 minutes) could be explained by the presence of fine particles (chalcopyrite), which are difficult to collide with the generated bubbles. Thus, longer grinding time should be recommended when the main objective is to depress the unwanted minerals more efficiently, even if it means a lower copper recovery. If the objective is to obtain a higher copper recovery, a shorter grinding time is recommended. The results show that a high size reduction is needed for this type of ore.

### Effect of depressant

In this study, the depressant was added during the grinding stage. Other addition points could have been studied, but Almina, Minas do Alentejo SA, determined that the grinding stage was the best point for the depressant addition. The first test was conducted to examine the influence of CaO dosage, which acted as a pH controller and a depressant for gangue minerals, mainly pyrite. The remaining parameters were maintained at the same level: grinding time = 90 min; pH = 10.5; promoter = 30 g/Mg; frother = 7 g/Mg. CaO was tested with three different dosages between 2000 and 3000 g/Mg. CaO was added to the pulp in a solid form without dilution. The results are presented in Fig. 3 and Table 2.

As shown in Fig. 3 and Table 2, the results of tests IV and V were very similar. In test VI, a higher copper grade was achieved, although with lower copper recovery, due to lower weight recovery (concentrate yield). As the lime dosage increased, the depressing effect increased and caused an unwanted effect on depression of chalcopyrite. The decrease of lime content in the pulp caused the increase of Cu recovery along with the recovery of unwanted minerals in the flotation concentrate as

expected. Thus, selection of the best lime dosage depends on the process stage. If the flotation tailings are further proceeded to a scavenger stage, it would be possible to use a larger quantity of lime to produce the concentrate with higher Cu grade. Although, if the main objective is to produce final tailings, a small lime quantity is recommended in order to obtain higher recovery.

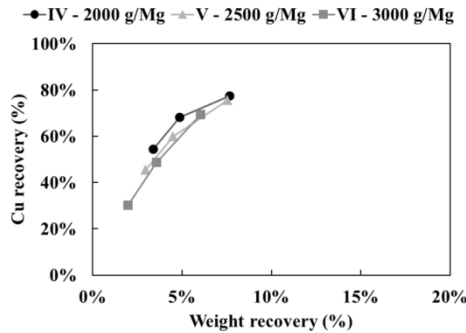


Fig. 3. Effect of CaO dosage on copper and mass recovery (Test IV – 2000g/Mg, Test V – 2500 g/Mg and Test VI – 3000 g/Mg)

Table 2. Recovery and grade of main elements (Test IV – 2000g/Mg, Test V – 2500 g/Mg and Test VI – 3000 g/Mg)

Test	Cu		Zn		Pb		Fe	
	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %
IV	15.62	77.33	1.91	9.42	2.54	35.06	34.30	6.92
V	15.16	75.56	1.51	7.55	1.73	23.49	34.68	7.03
VI	18.05	69.40	1.52	6.05	1.71	18.57	36.00	5.66

Sodium metabisulphite (MBS) (Fig. 4, Table 3) was used as the depressant for sphalerite. MBS was also added in the grinding stage at two different dosages: 750 (Test VII) and 1000 g/Mg (Test VIII). The remaining parameters were maintained at the same level: grinding time=90 min; pH=10.5; promoter=30 g/Mg; frother=7 g/Mg.

The test performed with 750 g/Mg (VII) of MBS showed lower Cu recovery than the test performed with 1000 g/Mg (VIII). This suggests that dosage lower than 1000 g/Mg of MBS is not efficient enough to achieve a good flotation performance, showing lower Cu recovery and grade. Test VIII showed a good balance between Cu recovery and selectivity. It means that 1000 g/Mg would be a good enough depressant dosage for flotation of the tested ore.

Finally, a mixture of lime and MBS was applied in order to take advantage of the effect of both depressants. The lime quantity was the same for both tests (2000 g/Mg) and MBS dosage was 500 and 750 g/Mg. The results are shown in Fig. 5 and Table 4.

The results from Test X showed higher copper recovery (87%) than Test IX (80%), however, TEST IX showed better depression of Zn and Pb. The results suggested that 2000 g/Mg of lime and 500 g/Mg of MBS would be recommended for a scavenger stage. The addition of MBS (MBS alone or mixed with lime) resulted in a higher Cu recovery value, and consequently, lower Cu grade. The test performed with lime alone produced better Cu grade, and higher efficiency to depress unwanted minerals, mostly Fe and Zn bearing minerals. If the objective is to efficiently depress Fe and Zn, lime should be used as the depressant. If the objective is to produce a final tailings in the rougher stage, i.e., if a scavenger stage is not present in the mineral process plant, the use of MBS is a good option because it maximizes the recovery of the valuable mineral with similar levels of depression for Fe and Pb. Otherwise, if a scavenger stage is the option in the plant flowsheet, the use of lime would be recommended.

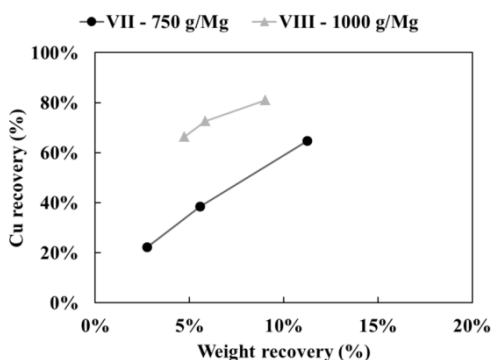


Fig. 4. Effect of MBS dosage on copper and mass recovery (Test VII – 750 g/Mg and Test VIII – 1000 g/Mg)

Table 3. Recovery and grade of main elements (Test VII – 750 g/Mg and Test VIII – 1000 g/Mg)

Test	Cu		Zn		Pb		Fe	
	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %
VII	9.10	64.68	1.95	14.23	0.81	15.98	37.84	10.89
VIII	14.04	80.84	2.77	16.13	2.16	34.27	34.20	7.98

Table 4. Recovery and grade of main elements (Test IX – 2000 g/Mg of CaO and 500 g/Mg of MBS and Test X – 2000 g/Mg of CaO and 750 g/Mg of MBS)

Test	Cu		Zn		Pb		Fe	
	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %
IX	11.13	80.28	1.89	13.43	1.89	36.31	36.23	10.27
X	9.57	86.73	2.23	20.01	1.99	48.60	36.48	13.15

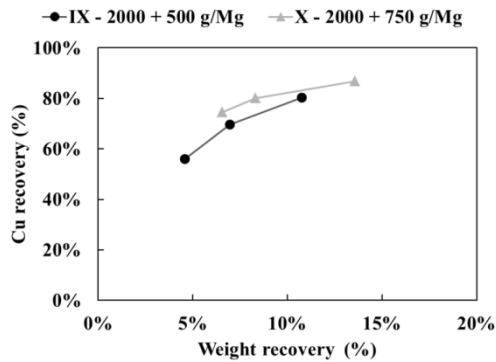


Fig. 4. Effect of CaO and MBS dosage on copper and mass recovery (Test IX – 2000 g/Mg of CaO and 500 g/Mg of MBS and Test X – 2000 g/Mg of CaO and 750 g/Mg of MBS)

### Effect of collector

For these experiments, the collector dosage varied between 20 and 45 g/Mg, while the remaining parameters were maintained at the same level: grinding time = 90 min, pH = 10.5, CaO = 2000 g/Mg, frother = 7 g/Mg. The results are presented in Fig. 6 and Table 5.

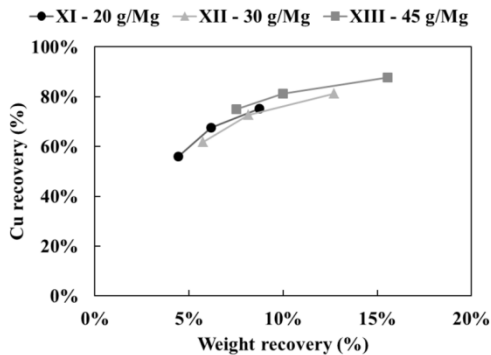


Fig. 5. Effect of collector dosage on copper and mass recovery (Test XI – 20 g/Mg, Test XII – 30 g/Mg and Test XIII – 45 g/Mg)

Figure 6 and Table 5 show that the Cu recovery increased with increasing the collector dosage, but consequently the Cu grade decreased. Test XIII showed higher Cu recovery, but also higher recovery of unwanted minerals, namely Pb-bearing minerals. Low collector dosage led to the decrease of Cu recovery and consequently to higher Cu grade. These results suggest that 20 g/Mg should be applied in the process if the objective is to produce higher concentrate grade in the rougher stage that would be followed by a scavenger stage. If the objective is to produce a final tailings in the



roughing stage, it is recommended to use 30 g/Mg of collector. Due to the higher recovery of Zn and Pb, a dosage of 45g/Mg should not be applied.

Table 5. Recovery and grade of main elements  
(Test XI – 20 g/Mg, Test XII – 30 g/Mg and Test XIII – 45 g/Mg)

Test	Cu		Zn		Pb		Fe	
	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %
XI	13.73	75.32	1.76	9.74	1.60	24.19	36.44	8.27
XII	10.09	81.20	1.95	16.18	1.83	41.17	37.60	12.30
XIII	8.57	87.77	2.35	24.05	1.79	50.74	37.28	15.16

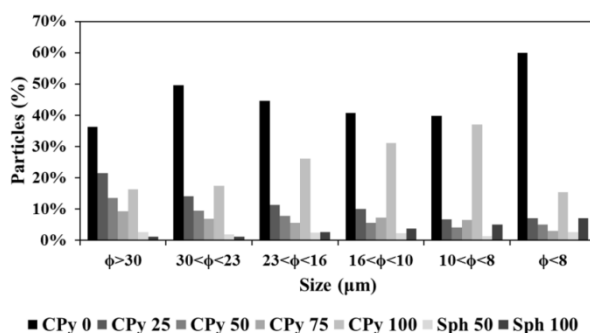


Fig. 6. Grade histogram conditional on size – TestXII

### Mineral liberation study

The objective was also to identify a new strategy that could improve the results obtained for Test XII, which obtained a good Cu recovery. For this purpose, the Cu concentrate was first hydrosized in a Cyclosizer into six size ( $\phi$ ) fractions, respectively  $\phi < 8$ ;  $8 < \phi < 10$ ;  $10 < \phi < 16$ ;  $16 < \phi < 23$ ;  $23 < \phi < 30$ ;  $\phi > 30 \mu\text{m}$ . Polished sections, belonging to each fraction, were analysed and grains were classified according to a pre-defined chalcopyrite/pyrite semi-quantitative bin scale of 0 CPy; 25 CPy; 50 CPy; 75 CPy; 100 CPy, which corresponds to approximately 0, 8.7, 17.3, 26.0 and 31.2 of Cu, respectively. In addition, particles that display 50 and 100 % of sphalerite were counted. This procedure was applied for the classification of 1000 grains from each size fraction in order to obtain the particle grade histograms conditional on size, which are shown in Fig. 6. It appears that pyrite liberated particles (0 CPy) are predominant in all the six concentrate size classes and are roughly between 40 and 60. It justifies the difficulty that floatation would have to improve the concentrate Cu grade. In fact, pyrite liberated particles should not be present in the concentrate, because the reagent strategy was supposed to depress these particles.

### Effect of frother

Assuming that the presence of pyrite particles in the concentrate could be due to mechanical flotation (entrainment), a new test with an increased frother dosage was carried out in order to improve pulp drainage around bubbles in the froth. In this flotation test (Test XIV), 14 g/Mg of frother was used (2-fold higher than in the Test XII), while maintaining the other parameters as before: grinding time = 90 min; pH = 10.5; CaO = 2000 g/Mg; promoter = 30 g/Mg). The results are presented in Fig. 8 and Table 6.

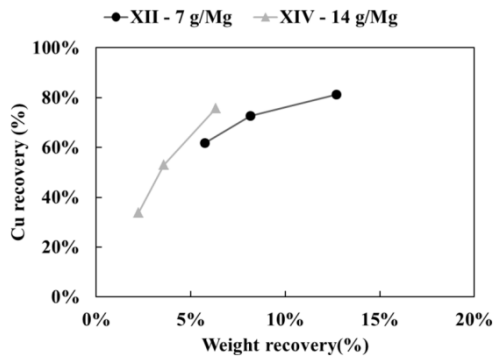


Fig. 7 - Effect of frother dosage on copper and mass recovery (Test XII – 7 g/Mg and Test XIV – 14 g/Mg)

Table 6. Recovery and grade of main elements (Test XII – 7 g/Mg and Test XIV – 14 g/Mg)

Test	Cu		Zn		Pb		Fe	
	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %	Grade %	Recovery %
XI	13.73	75.32	1.76	9.74	1.60	24.19	36.44	8.27
XII	10.09	81.20	1.95	16.18	1.83	41.17	37.60	12.30
XIII	8.57	87.77	2.35	24.05	1.79	50.74	37.28	15.16

Results shown in Fig. 8 and Table 6 indicate that Test XIV achieved a better balance between copper and unwanted minerals recovery. Both recovery of Cu and unwanted minerals recovery decreased indicating that the concentrate with higher copper grade was collected. This could possibly happen due to the improvement of frother stability, which reduced entrainment of fine particles (Smith and Warren, 1989). Therefore, the frother dosage increase could reduce entrainment and contribute to an increased efficiency in flotation, reducing the copper recovery due to non-flotation of free chalcopyrite particles with sizes lower than 8  $\mu\text{m}$ , but also reducing the unwanted minerals recovery. Finally, increasing the frother dosage reduced the flotation rate, therefore, this reduction could be due to a slower process of particles classification into the froth phase. This would improve the precision of this stage and

slow down the recovery ratio. If the flotation time was extended for Test XIV, Cu recovery could be higher than the obtained in Test XII, and ultimately lead to a higher grade.

## Conclusions

The effects of mineral liberation and chemical reagents dosage were assessed for the froth flotation of a complex sulphide ore from the Aljustrel mine. The process showed increased susceptibility for variation of grinding time. Depression of unwanted minerals improved significantly for longer grinding times, however the copper recovery also decreased. Reduction in the particle size was required as a consequence of the high complexity of the ore. CaO and MBS played a role of depressants of unwanted minerals and improved the concentrate grade. The results suggested that froth flotation was less sensitive to the variation of CaO dosage. High CaO dosages could cause depression of chalcopyrite. In the case of MBS addition, the study of optimal dosage should be conducted in a careful way because low variations could result in higher process changes. Concerning the mixture of CaO and MBS, the chemical reaction on the mineral surface should be examined in order to understand the compatibility of these two compounds and to predict efficient mixture dosages. It was shown that the collector played a key role in controlling the selectivity of Zn and Pb. It was also shown that the concept of increasing collector dosage to improve metal recovery had limits, mainly due to the increase of unwanted minerals recovery. It was also shown that for complex sulphide ores, the frother dosage played an important role due to the increasing stability of froth phase and controlling entrainment of unwanted particles. The froth phase analysis could be an important contribution, suggesting that the addition of frother could lead to a more efficient selective process at the Aljustrel Plant.

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