RECOVERY OF SULPHIDES AS BYPRODUCTS FROM A GOLD FLOTATION CIRCUIT[1]

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Abstract

This study was performed as part of a wider project conducted by *Kinross* Gold Corporation on the desulfurization of sulphide gold ore tailings from the Morro do Ouro mine. The ore processed generally contains 3% to 5% sulphides distributed along the rock foliation and concentrated in quartz boudins. A study of the liberation of sulphides in the industrial floatation circuit concentrate was conducted by scanning electron microscopy with a second-generation automated mineralogy system (MLA - Mineral Liberation Analyzer). For chalcopyrite, liberation from the gangue minerals reached values higher than 92%, and for sphalerite, higher than 96% in all fractions. In contrast, for 100% liberated minerals of interest, the values obtained in all fractions were lower than 0.52% for chalcopyrite, 0.65% for galena and 0.28% for sphalerite, showing how low the levels of these minerals in the concentrates are when compared to pyrite and arsenopyrite. The use of Aero 3894 and 3418A collectors allowed selectivity in the recovery of chalcopyrite, galena and sphalerite in the three rougher floatation cells. In the copper rougher cell, we obtained recovery figures higher than 79% for chalcopyrite, lower than 19% for galena, sphalerite and pyrite, and lower than 10% for FeAsS. In the Pb rougher cell, the recoveries obtained were higher than 68% for galena and lower than 19% for sphalerite and 11% for chalcopyrite and FeAsS. However, the highest recoveries in all three cells were obtained for pyrite, corresponding to 30%.

Key words: Flotation; Sulphide Minerals; Liberation; Collectors.

RECUPERAÇÃO DE MINERAIS SULFETADOS COMO SUBPRODUTOS PROVENIENTES DE UM CIRCUITO DE FLOTAÇÃO DE OURO

Resumo

Neste trabalho apresenta-se um estudo que faz parte de um projeto mais amplo da *Kinross* Gold Corporation para a dessulfurização dos rejeitos gerados a partir do minério aurífero sulfetado da mina Morro do Ouro. O minério processado apresenta, em geral, entre 3% a 5% de sulfetos, disseminados ao longo da foliação da rocha e concentrados nos boudins de quartzo. Um estudo da liberação dos sulfetos que constituem o concentrado de flotação do circuito industrial foi realizado por microscopia eletrônica de varredura com mineralogia automatizada de segunda geração (MLA - *Mineral Liberation Analyzer*). Para a calcopirita, a liberação da ganga atinge valores acima de 90%, para galena acima de 92% e para esfalerita acima de 96%, em todas as frações. Por outro lado, os minerais de interesse 100% liberados, atinge valores abaixo de 0,52% para calcopirita, 0,65% para galena e 0,28% para esfalerita, em todas as frações, evidenciando os baixos teores dos mesmo nos concentrados, quando compardos à pirita e arsenopirita. O emprego dos coletores Aero 3894 e 3418A permitiu uma seletividade na recuperação de calcopirita, galena e esfalerita nas três etapas rougher de flotação. Na etapa rougher de cobre, foram obtidas recuperações acima de 79% de calcopirita; menores de 19% para galena, esfalerita e pirita; e menores de 10% para FeAsS. Na etapa rougher Pb, foram obtidas recuperações acima de 68% de galena e menores de 19% para esfalerita e 11% para calcopirita e FeAsS. Porém, apresentou as maiores recuperações de pirita dentre as três etapas, correspondendo a 30%.

Palavras-chave: Flotação; Minerais sulfetados; Liberação; Coletores.

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1 INTRODUCTION

Activities related to the production of minerals generate a significant volume of mineral mass that is rejected in the mining and beneficiation processes.$^{(1)}$ The tailings from ore processing units contain a large quantity of solid particles in suspension, heavy metal ions, floatation reagents, organic matter etc.$^{(2)}$
The tailings from the industrial floatation circuit in plant 2 at *Kinross Brasil Mineração S/A* are discharged into the tailings dam at a rate of 41 Mt/year. Studies are being conducted on desulphurizing the tailings and producing differentiated sulphide concentrates from B-2 ore containing 3% to 5% sulphides distributed along the rock foliation and concentrated in quartz boudins. The aim of these studies was to recover byproducts and reuse the tailings generated by the process with a view to reducing the size of tailings disposal areas. The aim of our study was to evaluate the performance of reagent systems for the selective floatation of sulphides (copper, pyrite, zinc and lead) from concentrates collected in the new plant at *Kinross* Paracatu. Over the last few years, many studies have been conducted using chelate reagents for sulphide floatation, although xanthates are still commonly used as collectors in floatation processes in the non-ferrous metals beneficiation industry. However, these collectors are active for the entire class of sulphide minerals and not specific to an individual mineral.$^{(3)}$
The sample examined was therefore subjected to mineralogical characterization tests and liberation studies, carried out by scanning electron microscopy with a second generation automated mineralogy system (MLA-Mineral Liberation Analyzer). The new microscope can be used for rapidly identifying and discriminating the mineral phases from backscattered electron images combined with EDS images (Energy-dispersive X-ray spectroscopy). The automated system produces relevant information on associations among the various mineral phases, and on liberation and particle size band.

2 MATERIALS AND METHODS

2.1 Reagents and Minerals

Samples of gold cleaner concentrate (approx. 150 kg) were collected in the floatation circuit and stored in a nitrogen atmosphere. The samples received were homogenized and subdivided into subsamples of approximately 1 kg, for use in the analysis and liberation studies and the floatation studies. Chemical analysis of the gold concentrates showed that the typical average feed composition is 7.2 g/t Au, 22.05% S, 6,147 g/t As, 152,014 g/t Fe, 1,392 g/t Cu, 1,715 g/t Zn and 3,382 g/t Pb. The P_{80} for the concentrate was approximately 38µm. The collectors evaluated were: Aero 5100 Promoter, Aero 3418A Promoter and Aero 3894 Promoter (Cytec Industries Inc.). The frother used was Flotanol (Clariant). In the sphalerite floatation stage, $CuSO_4$ 97% P.A. (Vetec) was used as an activator. Table 1 gives the average composition of the collectors evaluated.

Table 1. Average composition of collectors

	Reagents	
Collector	**Composition**	**% (w/w)**
Aero 5100 Promoter	Thionocarbamate	60-100
	Isobutanol	5-7.5
	Butanol	5-7.5
Aero 3418A Promoter	Sodium Di-isobutyl dithiophosphinate	50-52
	Triisobutylphosphinate	1.0
Aero 3894 Promoter	Isopropyl ethyl thionocarbamate	88-94
	Isopropanol	2-5

2.2 Sample Preparation and SEM/MLA Image Acquisition and Processing

During sample characterization by MLA, 700 g amounts were wet-classified to obtain the various granulometric fractions and determine the content levels for each size band. Amounts of approximately 5 g were obtained from the various fractions after homogenization and taken for producing the specimens. The specimens were prepared by adding approximately 12 g epoxy resin for each 5 g sample in a mould 30 mm in diameter, where it remained for 3 days (drying time). After drying, specimen surfaces were polished using Struers Tegra equipment and 75 µm, 15 µm, 9 µm and 6 µm abrasives, and the final finish was obtained using suspensions of 3 µm and 1 µm diamond to produce random slice surfaces. The polished specimens were then coated with carbon to produce images using the scanning electron microscope coupled to the x-ray dispersive energy system (SEM/EDS – FEI Quanta 400). The studies were conducted using the MLA system with MLA Suite 2008 software.

2.3 Floatation Tests

The floatation tests were run in an sub-aerated D12 Denver laboratory floatation cell at 1,500 rpm, on approximately 1 kg pulp with a concentration of solids between 30% and 40%. The pH was controlled using sodium hydroxide solutions (10%). First, the pulp was homogenized and adjusted to the required pH value. After adding the collectors (conditioning time = 1 min.) and the frother, air was introduced into the cell and the total floatation time was 5 mins. The floatation tests were run as shown in the flowchart in Figure 1. The floatation process selected was aimed at selectively recovering first the chalcopyrite, then the galena and finally the sphalerite. The tests were run in triplicate. The electrochemical potential (Eh) of the pulp was measured at all stages in the floatation tests (initial pulp Eh and Eh during the conditioning and floatation stages) using a saturated calomel electrode (SCE) and platinum indicator electrode.

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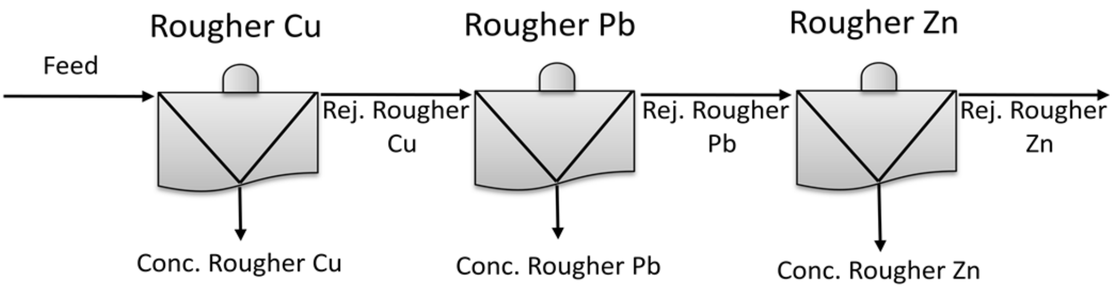

Figure 1. Floatation test circuit.

The pH for the chalcopyrite and galena rougher stages was 9.8 and the test conditions as follows: collector conditioning time of 1.0 min, collector concentration of 20 g/t, floatation time of 5 mins., Flotanol (10 g/t) frother. The pH for the sphalerite rougher floatation stage was 12. The process involved initial conditioning (2.0 min) with the $CuSO_4$ activator at a concentration of 200 g/t, a second conditioning phase with the collectors (1.0 min) at a concentration of 20 g/t and finally, floatation time of 5 mins. using Flotanol.

All the products obtained (tailings and rougher concentrates) were dry-filtered at 60°C and weighed for calculating the recovered mass. Portions of these products were sent to *Kinross* laboratories for chemical analysis of Au, As, Pb, Zn, Cu, Fe and S. The analytical data were used for calculating the metallurgical balance sheet.

3 RESULTS AND DISCUSSION

3.1 Mineral Liberation Studies

As with any mineral processing operation, floatation separation efficiency can be determined using various characteristics, many associated with the feed. Liberation is an essential factor that significantly affects recovery and product content figures, and if liberation is insufficient, this will limit the separation efficiency. It depends on the microstructure (texture) of the ore, in terms of particle size, valuable mineral grain size distribution, interactions and the bond between the minerals of interest and the gangue.(4)

Liberation analysis was conducted by counting particles and examining their respective compositions in terms of the mass of minerals of interest, chalcopyrite, galena and sphalerite, in respect of liberation classes of 0% (gangue minerals), 0% to 100% (minerals of interest as a proportion of other minerals) and finally 100% particles (minerals of interest 100% liberated) in the various fractions.

Figure 2 shows mineral liberation as a function of mass percentage, per grade class evaluated. In Figure 2a, 2b and 2c the y-axis is arbitrarily cut into 8% for better visualization of the composites and chalcopyrite, sphalerite and galena particles, respectively. We can see clearly from Figure 2 that the 0% liberation class, i.e. gangue minerals, corresponds to the highest percentages in the composition of particles expressed in terms of mass, by comparison with all minerals of interest. For the chalcopyrite, the liberation of gangue minerals reaches values of over 90%, for galena over 92% and for sphalerite over 96%, in all fractions. In contrast, for 100% liberated minerals of interest, the values are below 0.52% for chalcopyrite, 0.65% for galena and 0.28% for sphalerite, in all fractions. These results represent extremely low contents of these minerals of interest (approximately 0.4% for chalcopyrite, 0.35% for galena and 0.22% for sphalerite) by comparison with other minerals.

Based on the results of distribution of the contents by mass of the particles in the different fractions shown in Figure 2, was calculated release of minerals of interest. In Figure 3 we can observe a similar behavior for the three minerals, ie, the fraction -38 +20 µm, are the highest values corresponding to 84.6% for chalcopyrite, 66% for galena and 88.6% for sphalerite, with the increase of the size particles, the liberation grade decreased for all evaluated minerals.

According to Olubambi et al.,(5) the efficiency of practically all mineral processing and hydrometallurgical operations is a function of the size of particle processed, due to the relatively high cost of reducing particle size and the problems associated with the separation of minerals. This means that higher or lower liberation are becoming more and more important. Data from the mineralogical association show that it is in these fractions of minerals of interest (chalcopyrite, galena and sphalerite) that they are associated with gangue minerals. Within these percentages of associated minerals, the main gangue mineral is pyrite, with figures of 55% for chalcopyrite, 79% for galena and 68% for sphalerite.

Figure 4 shows a synthesis of the results obtained for theoretical recoveries and contents of minerals of interest corresponding to chalcopyrite, galena and sphalerite in the concentrate obtained from the industrial floatation circuit, combining the different fractions.

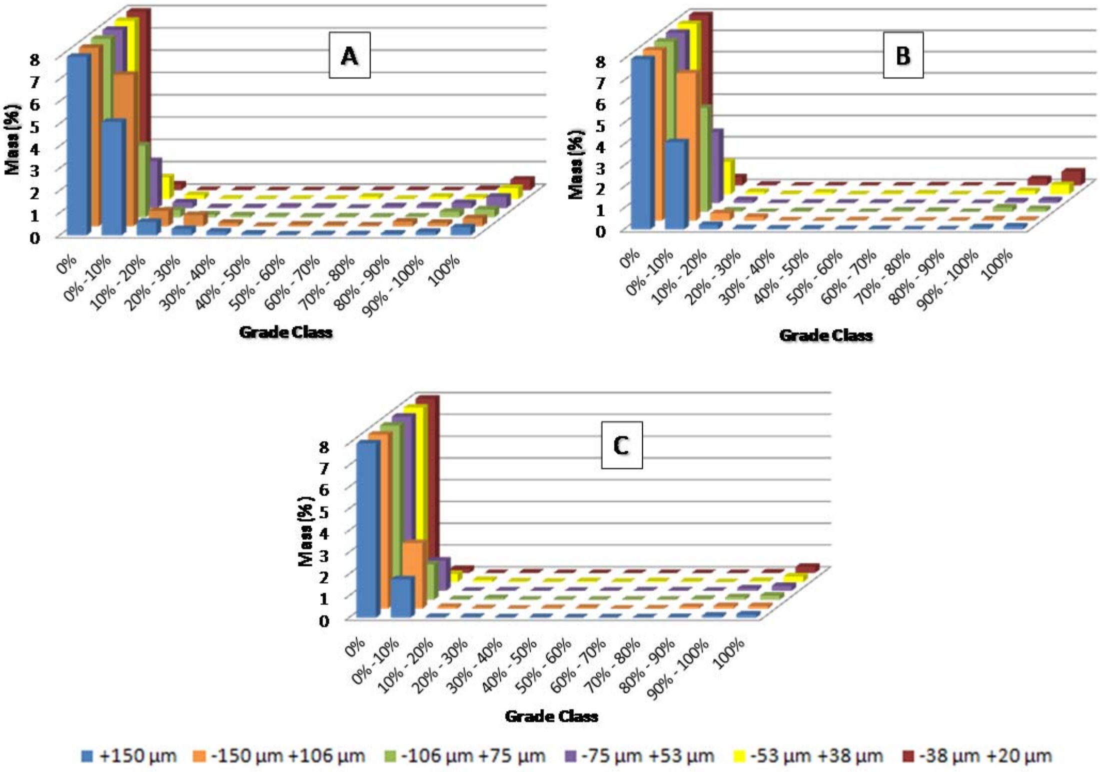

Figure 2. Distribution of particle grades, conditional by particle size. (a) Chalcopyrite; (b) Galena; (c) Sphalerite.

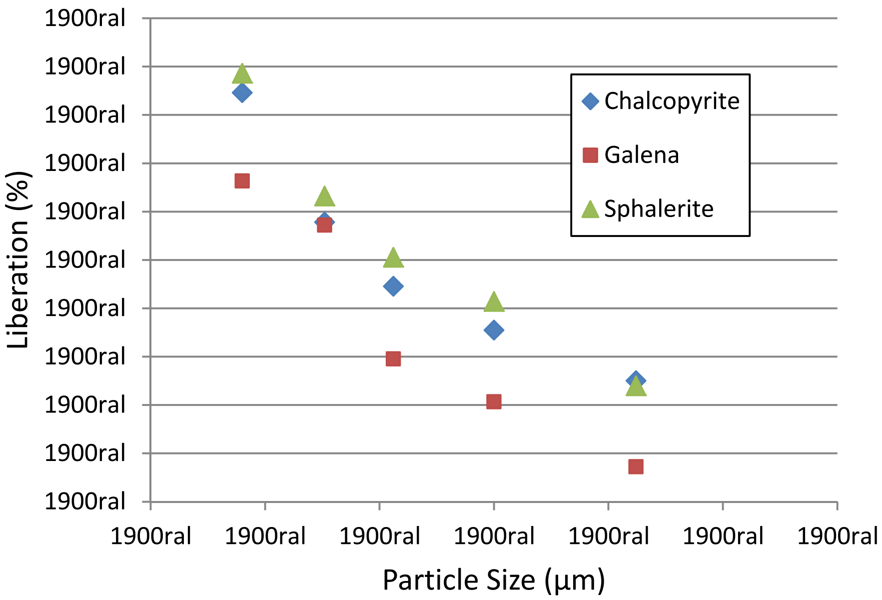

Figure 3. Liberation of particles, conditional by particle size.

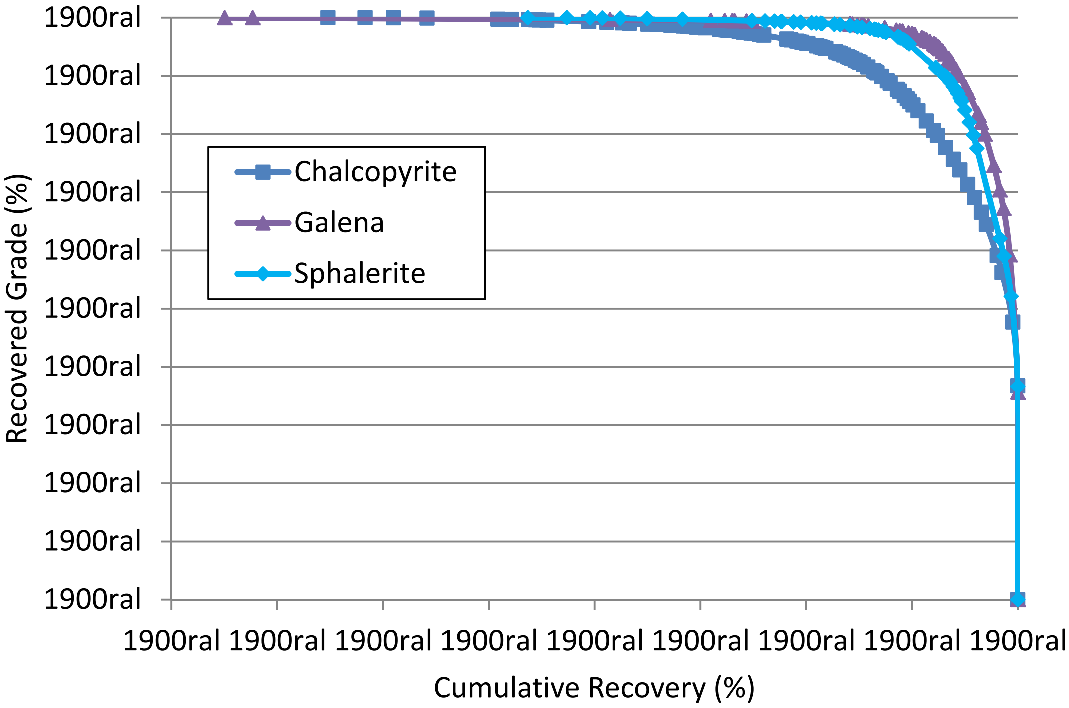

Figure 4. Theoretical recovery figures for contents of chalcopyrite, galena and sphalerite combining the different fractions.

3.2 Floatation Tests

Non-traditional collectors (i.e. non-xanthates) are being used in sulphide floatation due to the complex combinations of the majority of exploitable minerals.(6-8) Non-traditional collectors are chelate reagents whose structure contains donor atoms with a chemical affinity for specific metal ions, conferring high selectivity.

Figure 5 gives the recovery figures for minerals of interest ($CuFeS_2$, PbS and ZnS) and the main gangue minerals (FeS_2 and FeAsS) for the Aero 5100, Aero 3418A and

Aero 3894 collectors tested in the Cu rougher cell with the objective of identifying possible collector selectivity for minerals of interest.

It shows that Aero 3418A exhibited, on average, the highest recovery figures for $CuFeS_2$ and PbS (88.7% and 77.4%, respectively) and the lowest for ZnS (20%). However, it recovered more FeS_2 (43.2%) than the other collectors tested. Pecina-Treviño et al.(9) observed a similar phenomenon is their microfloatation tests. Using the same collector, they obtained high recovery figures for galena, but also high recovery figures for pyrite, suggesting that the pyrite surface was being activated by species of lead that appeared in solution as a result of the dissolution of Pb sites on the surface of the galena. These species, once adsorbed on the pyrite surface, produce an effect similar to that of the galena, promoting collector adsorption.

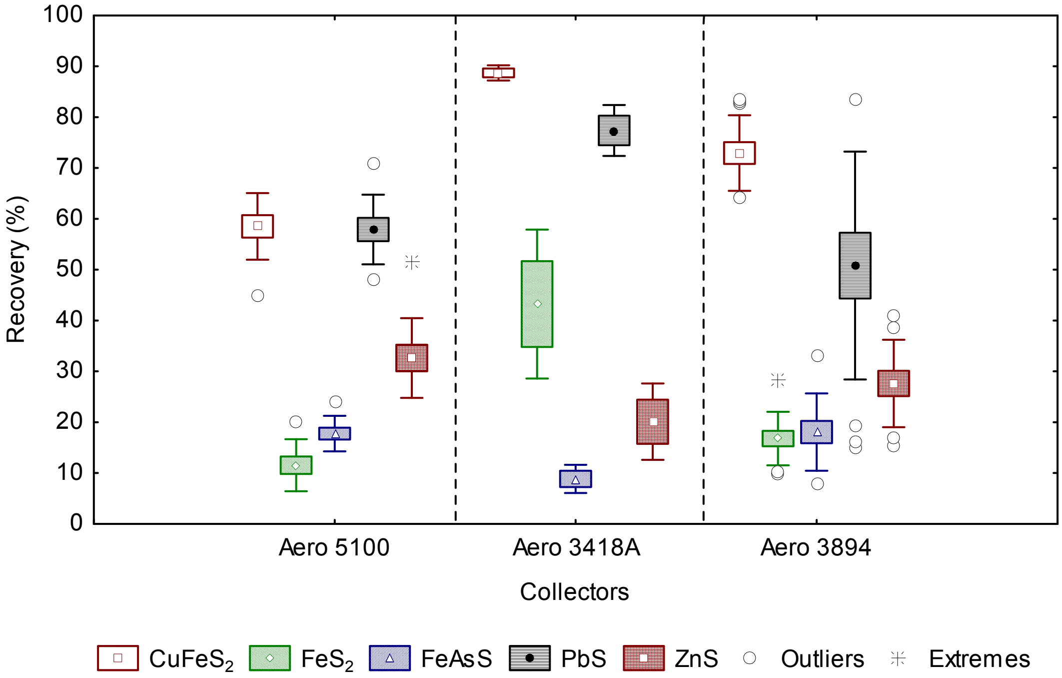

Figure 5. Recovery of minerals of interest and the main gangue minerals (pyrite and arsenopyrite).

The results of the tests conducted on the Aero 5100 collector show a more uniform recovery for both $CuFeS_2$ and PbS (58.5% and 57.9%, respectively), and the highest for ZnS (32.6%). Of the collectors tested, Aero 5100 produced low recovery figures for FeS_2 and FeAsS (11.5% and 17.7%, respectively). But Aero 3894, since it is a thiocarbamate with a longer alkyl chain, exhibited $CuFeS_2$ recovery of 73%, higher than Aero 5100. Recoveries of other minerals of interest, as well as gangue minerals, were close to those obtained using Aero 5100, corresponding to approximately 51% for PbS, 28% for ZnS, 16.8% for FeS_2 and 18% for FeAsS.

The nitrogen in thiocarbamates suggests a more covalent arrangement than the oxygen in traditional xanthates, but the phosphorus in the dithiophosphinates suggests an even more covalent arrangement in the functional group. According to Lotter and Bradshaw,(10) the differences in the behaviours of the collectors are attributable to the differences in the structure and consequent behaviour of the donor atom.

Oxygen has a single electron pair with more highly polarized atoms, and tends to be an electron donor, whereas nitrogen tends to be an electron acceptor. Therefore, the oxygen atom in the dithiophosphinate molecule has a strong electron-stripping effect. This, combined with the fact that phosphorus is more electropositive, suggests that dithiophosphinates are more selective collectors. However, an increase in the length of the alkyl chain (propyl to butyl) can reduce the solubility of the metal salt formed, making it stronger. For thiocarbamates, the N is less electronegative than the O, which has a greater tendency to donate electrons, resulting in a less selective, although stronger, collector. Based on the results above, we opted for the circuit in Figure 1 with addition of the Aero 3894 collector in the Cu rougher cell, the Aero 3418A collector for the Pb rougher cell, adding the Aero 3894 collector once again in the Zn rougher cell, since the sphalerite has already been activated by the copper sulphate.

3.3 Floatation Circuit Tests

Floatation is an industrial process for selectively separating valuable minerals from non-valuable minerals.(11,12) The characteristics of the particles to be processed (size, composition, hydrophobicity, hydrodynamic environment) and the chemical characteristics of the solution are decisive factors in good floatation.(13) Figure 6 illustrates a test conducted in triplicate, representing recoveries of minerals of interest ($CuFeS_2$, PbS and ZnS) and the main gangue minerals (FeS_2 and FeAsS) in the Cu, Pb and Zn rougher cells.

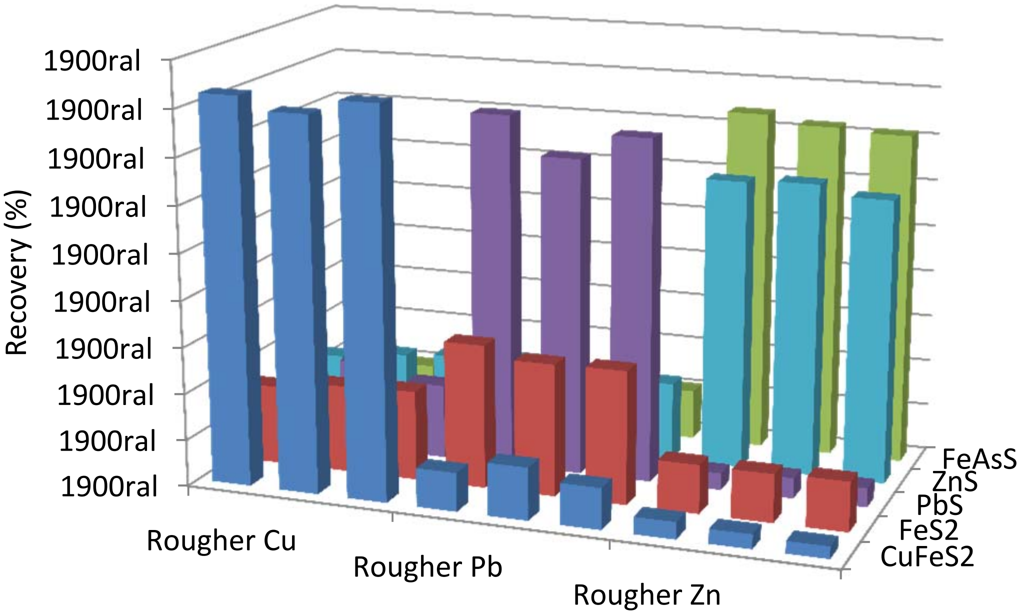

Figure 6. Recoveries of chalcopyrite, galena, sphalerite, pyrite and arsenopyrite in floatation circuit rougher cells.

As can be seen in Figure 6, the use of Aero 3894 and 3418A collectors in the sequences described above resulted in some selective recovery of chalcopyrite, galena and sphalerite in the three rougher floatation cells. In the Cu rougher cell, recovery of chalcopyrite was higher than 79%, lower than 19% for galena, sphalerite and pyrite, and lower than 10% for FeAsS.

In the Pb rougher cell, recovery of galena was higher than 68% and lower than 19% for sphalerite and 11% for chalcopyrite and FeAsS. However, the highest recoveries were for pyrite in all three stages, corresponding to 30%.

The concentration of 20 g/t of the collectors used could have affected recovery of both minerals of interest and gangue minerals. According to Johnson(14) in his studies on the liberation of sulphide minerals, the addition of the collector should be restricted to avoid undesired recovery of gangue minerals in intermediate fractions. However, recovery of minerals of interest liberated in fine fractions can drop in some systems, as a result of insufficient adsorption of the collector per unit area and/or excessive collection of activated species such as ferrous hydroxides.

The activation of zinc minerals, especially sphalerite, by the copper sulphate is already well known.(15) As we can see, in the Zn rougher cell with the addition of copper sulphate (200 g/t), there was a concentration of sphalerite that corresponded to recovery figures higher than 61%. However, the gangue minerals, pyrite and arsenopyrite, were recovered, with average figures of 10% and 71%, respectively. Similar results were obtained by Natarajan and Nirdosh,(16) who used a new collector (HCNPHA) for sphalerite, with recovery of approximately 80%. However, the pyrite was floatated with the spahlerite, a significant disadvantage. Shen, Fornasiero e Ralston,(17) studying the influence of pH on the selective recovery of sphalerite and pyrite, both oxidized, observed that in a highly alkaline medium (pH 11 to 12), the highest recoveries of sphalerite (> 85%) were achieved and the lowest of pyrite (< 20%). Wang, Forssberg e Bolin(18) showed that the copper adsorption rate resulting from activation is much faster for sphalerite than pyrite. In contrast to pyrite, arsenopyrite seems to be activated together with sphalerite, resulting in high arsenopyrite recoveries in a strongly alkaline medium.

4 CONCLUSIONS

The study of the liberation of sulphides using scanning electron microscopy with second generation automated mineralogy system (MLA-Mineral Liberation Analyzer) revealed that the liberation of gangue minerals in all fractions was as follows: chalcopyrite higher than 90%, galena higher than 92% and sphalerite higher than 96%. In contrast, 100% liberated minerals reached values lower than 0.52% for chalcopyrite, 0.65% for galena and 0.28% for sphalerite, in all fractions, showing how low the contents were in the concentrates, by comparison with pyrite and arsenopyrite. The use of Aero 3894 and 3418A collectors allowed some selectivity in the recovery of chalcopyrite, galena and sphalerite in the three rougher floatation cells. In the copper rougher cell, recoveries higher than 79% were obtained for chalcopyrite, lower than 19% for galena, sphalerite and pyrite, and lower than 10% for FeAsS. In the Pb rougher cell, recoveries higher than 68% were obtained for galena and lower than 19% for sphalerite and 11% for chalcopyrite and FeAsS. However, the best recoveries were for pyrite in all three cells, corresponding to 30%.

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