



# Modelling flotation per size liberation class – Part 1 – Minimizing the propagation of experimental errors in the estimate of flotation recovery



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## ABSTRACT

Mineral liberation has a strong effect on flotation performance. However, mainly due to stereological bias, the typical 2D scanning microscope mineralogical analysis is subject to limited representativeness and to high error propagation. In addition, grades calculated from the mineralogical distributions must be compatible with reconciled grades, estimated with mass balance calculations.

Despite the existence of several stereological correction methods, only few approaches have been developed to solve this integration issue. Beta reconciliation method is able to minimise errors in liberation data while reconciling balanced grades with the grades calculated via the distribution of liberation classes.

This is the first of a series of papers that aims at investigating and modelling flotation per size liberation classes. In this work, the beta adjustment method is detailed and optimized. Results from tests performed on a continuous chalcopyrite flotation circuit shows that the method provides the basis for a consistent balance, allowing to evaluate the flotation performance considering the size liberation classes.

## 1. Introduction

In terms of ore characteristics, mineral liberation is the parameter that establishes the maximum possible efficiency in any mineral concentration process involving only physical separation. Therefore, it is an essential criterion for designing new circuits, developing new processes and optimizing existing ones. This feature places mineral liberation as the central focus of modelling and simulation techniques. In recent years, the advance of automated mineralogy techniques has allowed detailed studies in several areas, such as:

- prediction of liberation spectra produced in mining and grinding (Mendez et al., 2009; Gay, 2004a and 2004b);
- design and optimization of flowsheets (Lotter et al., 2011; Dai et al., 2008)
- geometallurgical studies (Hunt et al., 2011; Parian et al., 2015)
- development of specific mathematical models (Zhang and Subasinghe, 2016).

Hence, representativeness of liberation data is fundamental. However, liberation data are distorted by error propagation. Two interrelated factors contributed to this: the representativeness of the analysed sample and the stereological bias.

When drawn from a continuous circuit, samples are subject to errors associated to process instabilities, sampling procedures, and physical and chemical analyses. These errors, which are commonly below 25%, can be minimized by appropriate process control, good sampling schemes and rigorous analysis methods, which can ensure that the samples represent the flows in the circuit.

In addition, liberation analysis requires specific sample preparation procedures. This analysis is performed per particle size fraction. To expose internal sections of the particles, for each size fraction, an aliquot is embedded in an epoxy block that is cut and polished to achieve scanning electron microscopy (SEM) analysis requirements. The automated mineralogy equipment analyses the mineralogical composition of a given number of particles which must be sufficient to provide a total composite grade compatible with the measured chemical grade of the sample (Savassi, 2006). This number is usually larger than 1000 in most studies performed since last decade.

In addition to that, the stereological bias - the difference between mineral distribution on a sectioned area basis (i.e 2D imaging) and the real volumetric distribution in the ore - may be a significant source of errors in 2D liberation analysis. Fig. 1 illustrates the effect of this bias.

This figure clearly shows that the areal grade determined by a 2D image can be completely different from the real particle grade, with biases higher than 100% in some liberation classes.

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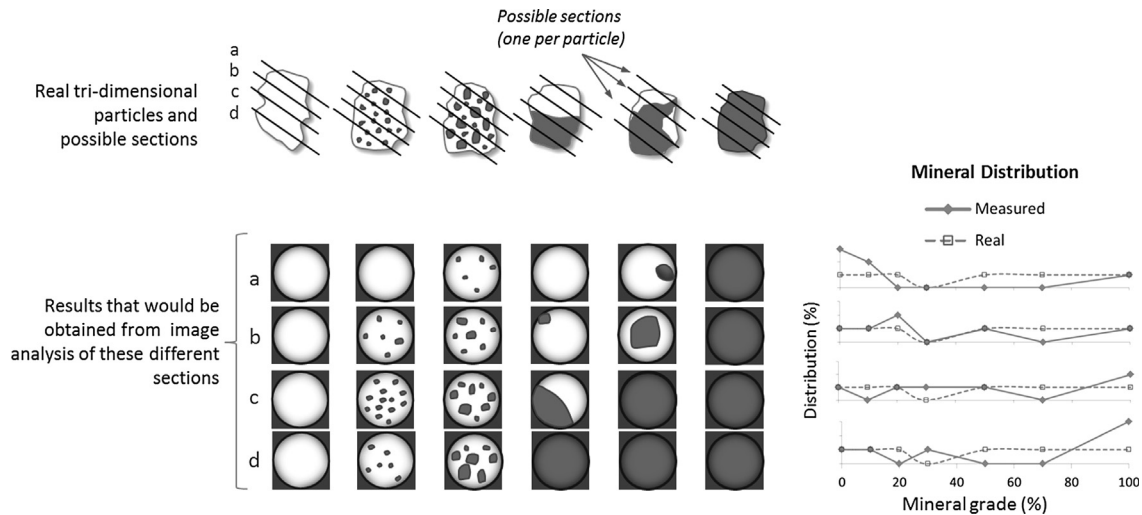


Fig. 1. Illustrative effect of stereological bias in liberation distribution ( ). adapted from Spencer and Sutherland, 2000

Stereological bias was first published by Gaudin (1939). With the assumption that the ore contained regular sized grains and constant grade, Gaudin defined a ratio between the areal and the volumetric liberation, called *locking factor*. Locking factors varied between 1.2 and 2.4, (Miller and Lin, 1988) what already demonstrated this bias could be as high as 140%. Petruk (1978) used a similar methodology.

Several other correction approaches emerged since then. Some methods involves the superimposition of more realistic texture patterns on the ore (Barbery, 1991; Gay, 1995). Others use a kernel transformational function to relate the liberation in the two different dimensions. (Miller and Lin, 1988; King and Schneider, 1997; Fandrich et al., 1998; Spencer and Sutherland, 2000). These methods are different in the way of processing the image analysis data and may estimate distinct volumetric distributions that may be more or less representative of the unknown 3D reality. However, in all of them, there were relevant differences between the estimated 3D distribution and the image analysis.

Also in most of these studies, there had been a concept that the analysis of a large number of particles would be able to reduce the stereological bias. If this was true, in the limit, it would mean that the 2D analysis could be used without correction provided a sufficient number of particles were measured; the issue would be to define this number.

However, the work of Ueda et al. (2016) presented a comprehensive evaluation of the effect of particle number in the liberation assessment and demonstrated that the dispersion of the degree of apparent liberation in 2D can be reduced by an appropriate number of particle section analyses, but the effect of stereological bias cannot be diminished. Fig. 2, extracted from this study, presents this effect.

Furthermore, Ueda et al. (2018) also confirmed that stereological bias is amplified in systems of multiple-phase particles or with relatively simple textures.

Therefore, in any quantitative study, it is essential to make a correction and estimate 3D data from 2D image analysis; 2D data sets that have not been corrected can result in misleading conclusions. Then, the issue is the choice of a correction method.

Every stereological or correction method must generate a 3D liberation distribution consistent with the sample chemical grade. This is a criterion to be fulfilled- or an objective function - in all approaches. This criterion has been considered sufficient when the liberation is analysed to provide information on a specific sample.

However, when evaluating the behaviour of a process per liberation classes, data from all flows must be consistent among themselves according to the circuit configuration. Nevertheless, this process consistency is only achieved after the mass balance reconciliation, which

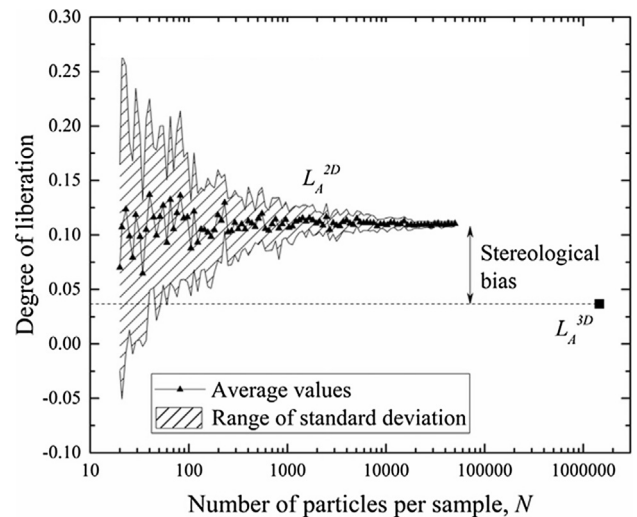


Fig. 2. Average values and standard deviations of the degrees of apparent liberation in 2D ( $L_A^{2D}$ ); for various number of particles (N) together with the degree of liberation in 3D ( $L_A^{3D}$ ); (from Ueda 2016).

adjusts and modifies all measured sample grades to reach values that represent the steady state process.

Therefore, stereological corrections that adjust the liberation data to the measured sample grades, most probably, will not be fully compatible to the reconciled sample grades of the mass balance. An integrated approach is necessary due to this interdependence of:

- measured grades – which represents the mineral proportion in the sample and in its size fractions;
- liberation – which represents the distribution of this mineral per classes in a sample size fraction, but is measured through 2 D image analysis, subjected to stereological biases;
- mass balance – which must represent the behaviour of this mineral in a steady state process, interconnecting different flows represented by samples and their fractions, whose grades must be mathematically adjusted to reflect the stable regime.

The beta reconciliation method developed by Savassi (2006) focuses on the whole reconciliation process involved in the metallurgical balance, instead of a stereological correction of each sample. The beta distribution function is used to estimate the 3D liberation distribution

from the 2D image analysis while reconciling each measured distribution with the balanced grade and density of the corresponding size fraction, in a hierarchical approach.

Another approach including liberation adjustment as part of a hierarchical mass balance procedure was developed in the study of [Lamberg and Vianna \(2007\)](#). The main difference between the two methods is on the reconciliation of liberation data with chemical grades. In [Savassi \(2006\)](#), the beta function is used to adjust the liberation distribution to the chemical grade of the size fraction. In [Lamberg and Vianna](#), this reconciliation was performed with a multiminerale ore through a series of steps involving (1) iterative adjustments of mineral grades from the mass balancing and liberation analysis; (2) grouping mineral phases and (3) normalization. [Savassi](#) used an ore containing one valuable mineral, galena, and assumed all other minerals as gangue, in a binary system.

These methods have the advantage of reducing the error propagation of the liberation data as well as performing the reconciliation of balanced grades with the grades calculated using the distribution of liberation classes.

In this study, the [Savassi](#) method was applied to balance a chalcopryrite flotation process. In a first trial, adjustment of all composite particles was very good but proportions of gangue had been overestimated in several cases. To solve this issue, the method was revised and optimized. This work revises the method, details its intermediate steps and adjusts the balance reconciliation of liberation classes to promote a more representative mass balance.

### 1.1. Beta distribution

The beta function - a distribution function developed in the 18th century - aims to estimate the real probability of an event from the observed frequency of the occurrence of this event in a given number of observations. The fact that the value of the real probability of an event would be achieved only in an infinite number of observations provides an idea of the amplitude of this inference. This ability makes the beta function very useful for several applications.

In mineralogical calculations, the beta function has been used for decades. [Barbery \(1991\)](#) and [King \(2001\)](#) reported its use as way of representing mineral particle liberation. Considering that the real mineral grade of particles,  $g$ , is the event to be reached in an infinite number of mineralogical determinations, the function can be written as shown in Eq. 1:

$$\text{Beta}(a, b) = \int_0^1 g^{a-1} \cdot (1-g)^{b-1} dg; a > 0, b > 0 \quad (1)$$

where  $g$  represents the mineral grade of the particle, or the average grade of a class of particles as a fraction in mass or volume, and  $a$  and  $b$  are the parameters that define the function. To adjust the function, it is necessary to obtain the parameters ( $a$  and  $b$ ) of a curve that best fits the liberation data from 2 D QEMScan analysis.

The beta function is defined within the interval  $0 < g < 1$ , without considering the extremes,  $g = 0$  and  $g = 1$ , equivalent to a grade equal to zero (gangue) or to 1 (fully liberated mineral), where the function is mathematically not applicable. Hence, in strictly speaking, the beta function could only be used for composite particles. A mathematical artifice must be used to solve this restriction.

## 2. Details and optimization of Savassi method

[Table 1](#) summarizes the main steps and tasks in the reconciliation method.

### 2.1. Stage 1 – Sampling

In this approach, sampling campaign must be rigorously planned and conducted to ensure maximum sample representativeness and to

minimise errors in the global balance. Preferably, both mass flowrates and grades should be measured, aiming at a balance with maximum redundancy and low biases.

### 2.2. Stage 2 – Converting chemical grades into mineralogical grades

When a chemical element is part of the composition of a single mineral, the calculation is stoichiometric. In this case, and if mass flow rates are also measured, this conversion can be made before or after the global balance, if only this valuable mineral is of interest.

For ores in which the valuable metal is part of the structure of more than one mineral or when there is an interest in obtaining the full mineralogical balance (including gangue), or when some flow rates need to be determined using grades, it is essential to perform the conversion into mineral grades before any level of balance reconciliation. The work of [Whiten \(2007\)](#) presents different ways of calculating the conversion for multiminerale ores.

### 2.3. Stage 3 – Global mass balances: water, mass and grades

Global (or total or unsized) ore mass and water balances are obtained with a mass balance reconciliation software. In the case of this study, this step was performed using both the ore flow rates and mineral grades as inputs and the estimate of errors of each of these measurements.

If:

- $M_{gl}$  = total measured mass flow rate of solids in each stream
- $g_{m,gl}$  = mineral grade in total mass of solids in each stream, obtained in the previous stage.
- $W_{gl}$  = total flow rate of water in each stream
- and the estimated errors of each of these measurements,  $\Delta M_{gl}$ ,  $\Delta g_{m,gl}$ ,  $\Delta W_{gl}$

then, global balances will generate balanced mass flow rates of ore and water as well as chemical grades. All global mass balance flows are identified by the subscript ( $_{gl}$ ). There will be the following outputs:

- $M_{gl}^*$  = balanced total flow rate of the mass of solids in each stream
- $g_{m,gl}^*$  = balanced grade of mineral *mrl* in each stream
- $W_{gl}^*$  = balanced flow rate of the water in each stream
- Residuals of the reconciliation of these parameters

where the symbol (\*) indicates that the data was adjusted by the reconciliation of the global mass balance, representing the “bulk” steady state. Because this is the first level of balancing, it is essential that the results are consistent and present the smallest possible residuals.

### 2.4. Stage 4 – Balances per size fraction and mineral grade per fraction

In the mass balance per size fraction, the balanced values obtained in the global reconciliation must be kept constant or include minimum alterations.

Within a reconciliation software, maintaining the values of global balance corresponds to assigning minimum deviations to these values. For the measured mass distribution in particle sizes and their respective grades, the deviations are established according to the estimated error in each fraction.

Then:

- $ms_i$  = measured percentage in mass of the size fraction  $i$ , resulting from particle size distribution analysis, from  $i = 1$  up to the total number of size fractions in the analysis,  $ng$
- $g_{msi}$  = grade of mineral *mrl* in the mass of solids of the size fraction  $i$ , obtained from the conversion of the chemical analysis per size

**Table 1**  
Main steps of Savassi method.

Stage	Task
1. Sampling	Guarantee a stable process and a rigorous sampling campaign to minimise deviations in the global balance
2. Conversion of chemical grades into mineral grades	Calculate the <i>mineral</i> grades of all flows and samples from the <i>chemical</i> grades (for valuables distributed in more than one mineral, it is important to perform this step before the mass balance)
3. Global water, mass and metal balances	Reconcile the total flow rates of solids and water as well as the chemical/mineralogical grades of each flow.
4. Balance per size fraction	Reconcile the flow rates of solids and the mineral grades in each size fraction in each flow. The results of global balances should be a constraint. In other words, the weighted sum of the reconciled grades and masses per size fraction should generate the values previously obtained in the global balance data.
5. Adjusting liberation data with a beta distribution	Adjust the liberation data of each size fraction to a beta curve so that this curve can be the most representative of the mineralogical data and present a sum of masses and mineralogical grades compatible with the balanced grades of the size fraction
6. Balances per liberation class	Reconcile the mass and liberation data of each liberation class in each size fraction of each flow for the whole process.

fraction to the mineral grade, from  $i = 1$  to  $ng$ .

- the estimates of deviations of each data,  $\Delta ms_i$ ,  $\Delta g_{ms_i}$
- In addition to residuals, this stage will generate the following main results:
  - $ms_i^{**}$  = balanced percentage in mass of the size fraction  $i$ , from  $i = 1$  to  $ng$ .
  - $g_{msi}^{**}$  = balanced grade of mineral *mrl*, in the mass of solids of the size fraction  $i$ , from  $i = 1$  to  $ng$ .

and, the reconciliation process of these values must be subject to the condition that the sum of reconciled grades of the size fractions is equal or the closest possible to the balanced global grade for each stream ( $g_{mgl}^*$ ). Mathematically, this means to impose Eq. (2) as a restriction.

$$\sum_{i=1}^{ng} ms_i^{**} \cdot g_{msi}^{**} = g_{mgl}^* \quad (2)$$

If the balance is dealing with *both* measured *masses* and measured *grades*, to converge, the reconciliation process per size fraction will need to perform small adjustments in the balanced global data. These new values of global data, considering the imposed restriction, must be very close to the results of the global balance. Therefore, this process will also include the following results:

- $M_{gl}^{**}$  = balanced global mass flow rate of solids in each flow, minimally adjusted by the reconciliation per size fraction.
- $g_{mgl}^{**}$  = balanced mass grade of mineral *mrl* in the solids of each stream, minimally adjusted by the reconciliation per size fraction.

### 2.5. Stage 4 – Reconciliation of liberation data

Automated mineralogy usually determines the distribution of liberation classes per size fraction. Each size fraction  $i$  is distributed in up to 12 classes with a volumetric or mass mineral grade -  $g_{v_j}$  or  $g_{m_j}$  - and each liberation class  $j$  contains a mass proportion  $f_{ml}$  of the total amount of analysed particles, equivalent to a volumetric proportion  $f_{vl}$ . In this work, the volumetric grades  $g_{v_j}$  will be considered as the basis for starting the reconciliation process for the liberation classes.

Eleven liberation classes were used for each size fraction. The first class,  $j = 1$ , is equal to the liberated gangue ( $g_{v,1} = 0\%$ ), while class 11 corresponds to the blend of completely liberated chalcocopyrite and almost completely liberated particles ( $90\% < g_{v,11} \leq 100\%$  mineral). The definition of this class that contains both liberated and almost liberated particles is important for the method, as will be explained later. Composite particles (or middlings) are associated to intermediate classes, with volumetric grades that vary in intervals of 10 percentage points ( $0,1\% < g_{v,2} < 10\%$ ;  $10\% < g_{v,3} < 20\%$ ; .....  $90\% < g_{v,11} \leq 100\%$ ). To compare the mineral grades obtained from the liberation analysis with grades determined by chemical analysis, it is necessary to convert both, the limits of classes as well as the average grade of each class, into mass grades  $g_m$ .

Each class  $j$  has a lower limit for the mineral grade,  $lw_j$ , and an upper limit,  $up_j$ . The lower limit of the class,  $lw_j$  is equal to the superior limit of the precedent class,  $up_{j-1}$ . For example, class  $j = 4$  presents  $lw_4 = 20\%$  ( $= up_3$ ) and  $up_4 = 30\%$ . Each class  $j$  has an average grade  $\bar{g}_{vlj}$  between  $up_{j-1}$  and  $up_j$ .

The volumetric grade of the  $i$ -*eth* size fraction, calculated from this liberation distribution is then given by the sum:

$$\bar{g}_{vsi} = \sum_{j=1}^{11} (\bar{g}_{vlj} \cdot f_{vlj}) \quad (3)$$

The grade calculated by this equation must be compared to the one obtained by the balance for the size fraction  $i$ . Once the grade from the balance is obtained from a chemical analysis and is equivalent to a mass grade, it is necessary to convert this volumetric grade into a mass grade, what can be made using the mineral and sample densities. The average grade in mass of each mineral class  $\bar{g}_{mlj}$  is obtained using Eq (4).

$$\bar{g}_{mlj} = \bar{g}_{vlj} \cdot \frac{\rho_{mineral}}{\rho_{sample}} \quad (4)$$

The grade of each size fraction will be calculated from the liberation data using Eq. (5). If  $f_{mlj}$  is the proportion in mass of each liberation class  $j$  for a particle size  $i$ , then:

$$\bar{g}_{msi} = \sum_{j=1}^{11} (\bar{g}_{mlj} \cdot f_{mlj}) \quad (5)$$

This average grade obtained by the weighted sum of the liberation classes will be distinct from the average grade obtained from the balance per size fraction. The objective of the interpolation is to reconcile the liberation distribution with this grade obtained in the balance, such that the adjusted grade  $g_{msi}^{***}$  is equal to the mineral grade of the size fraction  $i$ , calculated using the size balance reconciliation,  $g_{msi}^{**}$ :

$$\bar{g}_{msi}^{***} = g_{msi}^{**} \quad (6)$$

This adjusted grade will be the sum of the adjusted grades in each mineral class, weighted by its proportion:

$$\bar{g}_{msi}^{***} = \sum_{j=1}^{11} (\bar{g}_{mlj}^{***} \cdot f_{mlj}^{***}) \quad (7)$$

where the symbol  $***$  indicates the adjusted value of the data in the level of the liberation class. In addition, based on the conservation of masses, the sum of the adjusted fractions needs to be equal to 1 (or 100%):

$$\sum_{j=1}^{11} f_{m,j}^{***} = 1 \quad (8)$$

Therefore, the problem consists of determining the adjusted values of fractions  $f_{m,j}^{***}$  and the adjusted grades  $\bar{g}_{mlj}^{***}$  to attend the restriction of mass conservation (eq. (8)), using the beta curve, in association with other parameters.

The calculation starts with an estimate of a beta curve, using initial values for the parameters  $a$  and  $b$ . Least squares estimates are used to adjust the parameters aiming at minimizing the difference among:

- this estimated beta curve and the data measured by QEMScan
- the grade determined with this estimated distribution and that obtained by the mass balance per size fraction.
- gangue determined by the estimated curve and the analysed gangue

Therefore, the adjusted parameters obtained with this method, mathematically, are the ones that best represent the sample, allowing that the calculated grade by the beta curve be the closest possible to the balanced data and, at the same time, to the sampled data.

However, before initiating this calculation, it is necessary to solve a mathematical issue. The beta function is not applicable to extremes. It is not defined for zero or one ( $g_1 = 0$  and  $g_{12} = 1$ ). Liberation distributions have a gangue class ( $g_1 = 0$ ), which is essential, and usually include one class with only fully liberated particles ( $g_{12} = 1$  or 100%), that is, there is a distinction between the class of almost liberated particles ( $90 < g_{11} < 100\%$ ) and the fully liberated particles ( $g_{12} = 100\%$ ). To use the beta function, it would be necessary to remove both, the gangue particles and the fully liberated particles, based on the original method. After this removal, the beta function could be used to adjust the composite particles and, then, the gangue and liberated particles could be reinserted into the distribution in a separate reconciliation.

The use of this original approach worked in most cases. However, in several cases, the removal of both extremes of the liberation distribution transferred the stereological biases of composites to gangue or fully liberated classes. This effect could lead to a much higher proportion of gangue or liberated particles than would be reasonable, which would be incompatible with sampled data.

Aiming at optimizing the calculation, QEMScan was adjusted to indicate the data of fully liberated particles together with particle almost liberated, generating a class  $0,9 < g_{11} \leq 1$ . This way, it was necessary to remove only the gangue class from the distribution<sup>1</sup>.

The use of this highly liberated class improved the accuracy of the original method, once the liberated class is also reconciled by the beta function and only the gangue must be removed from this adjustment.

To remove the proportion of gangue in class  $j = 1$ ,  $f_{m,1}$ , it is necessary to normalize the distribution of composite classes ( $j = 2$  up to  $j = 11$ ) in a way to have a total sum of 100%. To do so, a new distribution  $f_{mmidd,j}$  is defined by:

$$f_{mmidd,j} = \frac{f_{m,j}}{1-f_{m,1}} \tag{9}$$

Then, it is possible to adjust a beta curve to this normalized distribution of composites.

The incomplete beta curve is used to perform this adjustment, where  $a$  and  $b$  are the parameters to be adjusted,  $up_j$  is the superior limit

<sup>1</sup> Although, in strict mathematical terms, it would be necessary to remove particles with grade equal to 100%, incorporating liberated particles to the precedent class allows the use of the beta curve for this new interval as if it was composite, that is, the value of  $g = 1$  becomes numerically inexistent but the real proportion of liberated particles is included in the class ( $90 < gv < = 100\%$ ). Before the incorporation of the fully liberated particles, the class ( $90 < gv < 100\%$ ) would have a theoretical average grade of around 95%. With the inclusion of liberated particles, the calculated grade of the new class with the beta function will be higher, around 97–98%, which mathematically allows the use of beta, and physically, includes the liberated class. The same approach cannot be used for the gangue, because the proportion of gangue is significantly high for several streams. Incorporating the proportion of gangue in the first class of mineralized particles would create an average grade too close to zero for this new class and the beta curve would not be applicable to adjust the data.

of class  $j$ , and  $F_{m\ midd,j}$  is the cumulative distribution of composite particles up to  $up_j$ .

$$F_{mmidd,j} = \text{Beta}(a, b, up_j), 2 < j < 11 \tag{10}$$

Since the beta function is defined as a standard formula in Excel and similar softwares, it is possible to use a regular spreadsheet to perform this calculus. To obtain a first beta distribution, it is necessary to estimate initial values for  $a$  and  $b$ , which will be subsequently adjusted. If these initial values are called  $a_{ini}$  e  $b_{ini}$  then:

$$F_{mmidd,j}^{initial} = \text{Beta}(a_{ini}, b_{ini}, up_j), 2 < j < 11 \tag{11}$$

From this initial cumulative function, a simple initial distribution  $f_{mmidd,j}^{initial}$  can be obtained for each class  $j$ :

$$f_{mmidd,j}^{initial} = \text{Beta}(a_{ini}, b_{ini}, up_j) - \text{Beta}(a, b, up_{j-1}), 2 < j < 11 \tag{12}$$

This initial curve must be compared to the sampled data. Since the proportion of gangue,  $j = 1$ , had been removed from the sampled distribution, it is necessary to estimate the proportion of gangue to be added to the curve to obtain a complete initial distribution. This requires an initial estimate for the value of gangue,  $f_{m,1}^{initial}$  from a measured value of gangue proportion  $f_{m,1}$ . To this aim, an interpolation parameter,  $\gamma$ , is defined. In turn, this value of  $\gamma$  requires an initial value,  $\gamma_{ini}$ . Therefore:

$$f_{m,1}^{initial} = f_{m,1} \cdot (1 + \gamma_{ini}) \tag{13}$$

The estimated value of this parameter allows re-calculating the complete distribution curve, from  $j = 1$  up to 11. The value for  $j = 1$  is given by this equation, Eq (13). The values from  $j = 2$  to 11 are re-normalized using the value for  $j = 1$  and the initial simple distribution for composite particles  $f_{mmidd,j}^{initial}$ :

$$f_{m,j}^{initial} = f_{mmidd,j}^{initial} \cdot (1 - f_{m,1}^{initial}), 2 < j < 11 \tag{14}$$

With these values, it is possible to obtain the initial complete cumulative curve  $F_{m,j}^{initial}$ , based on the beta curve:

$$F_{m,j}^{initial} = f_{m,1}^{initial} + \sum_2^j f_{m,j}^{initial} \tag{15}$$

To determine the values of  $a$ ,  $b$  and  $\gamma$  the method of least squares is used. In the original method (Savassi, 2006), the sum of the following values was minimized:

1. The difference between the distribution of composite particles calculated by the beta curve  $f_{mmidd,j}^{initial}$  and the measured distribution of composite particles,  $f_{mmidd,j}$  divided by the number of composite classes.

$$S_1(a, b, \gamma) = \sum_{j=2}^{j=11} (f_{mmidd,j}^{initial} - f_{mmidd,j})^2 / n \tag{16}$$

2. The difference between the cumulative distribution of composite particles calculated by the beta curve,  $F_{mmidd,j}^{initial}$  and the cumulative distribution of measured composite particles,  $f_{mmidd,j}$ , divided by the number of composite classes:

$$S_2(a, b, \gamma) = \sum_{j=2}^{j=11} (F_{mmidd,j}^{initial} - f_{mmidd,j})^2 / n \tag{17}$$

3. The difference between the balanced grade for the size fraction  $i$ ,  $g_{msi}^{**}$  and the grade calculated by weighting the average grades of each class and the distribution of the initial liberation  $\sum_{j=1}^{11} (g_{mj} \cdot f_{m,j}^{initial})$ :

$$S_3(a, b, \gamma) = \sum_{j=1}^{j=11} \left( g_{msi}^{**} - \sum_{j=1}^{11} (g_{mj} \cdot f_{m,j}^{initial}) \right)^2 \tag{18}$$

In this work, a fourth function was added, to guarantee that the proportion of gangue also had minimum deviation in comparison to the measured values, favouring lower deviation in terms of grades. This function is the difference between the proportion of gangue,  $f_{m,1}^{initial}$  using  $\gamma$  and measured gangue,  $f_{m,1}$ :

$$S_4(a, b, \gamma) = (f_{m,1}^{initial} - f_{m,1})^2 \quad (19)$$

Furthermore, as in this process of minimization, the most important aspect is to guarantee that the liberation grade is compatible with the size fraction grade, it was necessary to add a higher weight to the functions  $S_3$  and  $S_4$ . Therefore, a new objective function is defined by the sum:

$$S_1(a, b, \gamma) + S_2(a, b, \gamma) + \zeta S_3(a, b, \gamma) + \eta S_4(a, b, \gamma) \quad (20)$$

The values of weights  $\zeta$  and  $\eta$  will depend on the numerical values used in the distribution, if the values are from 0 to 100, as percentages, or from 0 to 1, as fractions. The use of these weights is important because they adjust the four factors to be in the same order of magnitude.

The minimization process is done via Solver in Excel and determines the adjusted values for  $a$ ,  $b$  and  $\gamma$ . With these parameters, the adjusted values of the simple and cumulative liberation distributions are determined.

## 2.6. Stage 5 – Balancing the liberation data

Once the liberation has been adjusted for all size fractions and for all flows, it is possible to perform the balance per size fraction. As most reconciliation softwares do not include this level of mass balance with redundancy, the calculation of this balance is distinct of the previous ones. The last level balanced via a reconciliation software was the size fraction. The beta curve adjusted the liberation data to reach the closest possible grades to the ones of the sized balance. However, to reconcile the balance, it is necessary that these grades are *equal*. Besides, each liberation class must have an average grade and a simple distribution in steady state, that is, each class must also have a reconciled balance among products. Fig. 3 represents these restrictions.

Considering these restrictions, there are two possibilities:

1. Calculate the balance using Excel spreadsheets, adjusting the data obtained in the beta curve in order to reach the reconciliation or
2. Use a reconciliation software to perform a complete balance for each size fraction, as if the mass of a single fraction, already balanced, would correspond to the total mass of a beneficiation plant. In this sense, it would be like if the size fraction, instead of being a second level of reconciliation, was the initial total mass, corresponding to the first level of balancing. Using this technique, the software can deal with liberation classes as a sublevel.

## 3. Tests

The basis of this work is the same as described in Santos et al. (2014). Three continuous pilot tests had been conducted at Vale Mineral Development Center, in a flotation mini pilot plant (MPP) using a chalcopyrite ore from an operating industrial plant in the north of Brazil. Each test had a specific froth height in the first rougher cell, to allow an evaluation of froth recovery. Tests were called Cal 01, Cal 02, and Cal 03 and were performed with low, intermediate, and high froth height, respectively.

The MPP is a continuous flotation setup, capable of reproducing industrial circuits. It comprises 12 cells of 1.7 L, similar to Denver laboratory cells, whose flows are interconnected by peristaltic pumps. By its compact form, the MPP enables good control of process variables, including air flow rate, pH, rotation speed and reagent dosage. Sampling can be performed by collecting the whole flow over long periods of time.

A simple circuit that included rougher, scavenger and cleaner stages with a feed of 110 g/min (7 kg/h), at 30% solids and with a particle size approximately 85 percent < 210  $\mu\text{m}$  was used. As this circuit aimed to evaluate the characteristics of flotation size-liberation class, there was no regrinding of intermediate concentrates, which would require a complete change in the particle size distribution and, therefore, a complete resetting of the model in the middle of the circuit. Tests were conducted in an open circuit to obtain a high accuracy in the parameters. It is important to clarify that it would not be difficult to achieve circuit stabilization with recirculation loads. However, flow rates are small in the MPP, only some grams per minute in intermediate flows. If circuit was closed, sampling of recirculating flows should be performed in small aliquots of 20 s taken every 10–15 min. Then, to obtain the appropriate amount of material to perform all the analyses, at least 150 g and ideally 400 g, the duration of each test and ore feed consumption would be multiplied several times, what would not be feasible due to the limited amount of ore available for these tests. With an open circuit, it was possible to collect the intermediate concentrates over the required time, around two hours, what reduced sampling bias and promotes accuracy.

Fig. 4 shows the flowsheet. Each square corresponds to a 1.7-L cell with a froth crowder, assembled to avoid stagnant froth zones in the cell. Froth crowder directs froth to the vicinity of the paddles. Crowder is a regular accessory of MPP and helps minimizing stabilization time for the pilot plant.

As collectors, sodium amyl xanthate and sodium isopropyl methylene thionophosphate were used. Ethyleneglycol-propylene oxide ether and MIBC, methyl isobutyl carbinol were used as frothers. Each reagent was added at a dosage of 10 g/t.

Due to the small flowrates, the whole flow of products were collected. Therefore, the obtained material was not a “sample” as a “portion” of the flow, but was the whole flow accumulated during a certain time. Tailings were taken in aliquots of 1 min. In particular, the whole flows of concentrates were accumulated for two and half hours. Considering that the residence time of total plant was 45 min (~15 min in Rg + Sc and ~30 min in Cl), concentrate flows were taken during 3 residence times without interruption. More than representative samples, these concentrates were consolidated products from the circuit. Flows of solids and water were determined with this whole amount. From the products, samples were split for chemical and physical analysis. Each test had a sampling campaign that lasted 5:30 h, divided in four blocks, as shown in Table 2.

These products were screened at 210  $\mu\text{m}$ , 150  $\mu\text{m}$ , 74  $\mu\text{m}$  and 44  $\mu\text{m}$  and the fraction below 44  $\mu\text{m}$  was analysed in a cyclosizer. Chemical analysis was performed for Cu, Fe, Si and S elements for the overall sample and for the size fractions.

The mineralogical analysis was conducted in QEMScan for size fractions of –210  $\mu\text{m}$  +150  $\mu\text{m}$ , –150  $\mu\text{m}$  +74  $\mu\text{m}$ , –74  $\mu\text{m}$  +44  $\mu\text{m}$  and –44  $\mu\text{m}$  +20  $\mu\text{m}$ , for the feed and the rougher 1 concentrate, rougher 2 tailings, scavenger concentrate, cleaner concentrate and cleaner tailings. The liberation distribution in other streams was obtained through the QEMScan estimate and mass balance. Fig. 4 illustrates the sampling points.

## 4. Results

### 4.1. Global balance

Global mass balance was calculated with the a reconciliation software, using all measured data as inputs: mass flow rates, pulp flow rates, solids percentages and copper grades. This redundancy increased the balance accuracy (Hodouin, 2010).

Balanced values and sampled data are shown in Table 3.

Reconciled values were very close to sampled data for all parameters, solids flow rates, pulp flow rates and copper grades. Almost all solid flow rates and copper grades presented low residuals, below 4%.

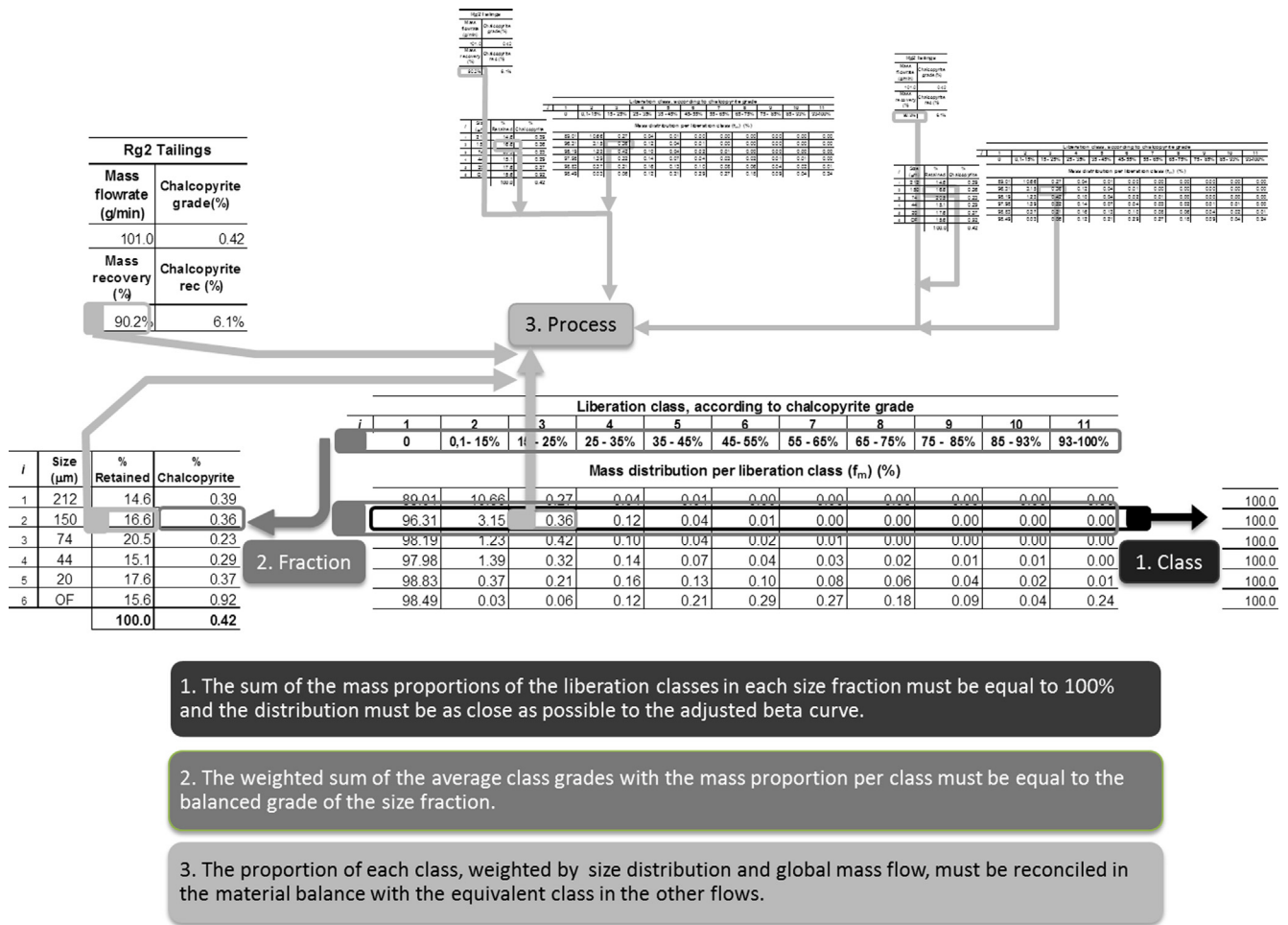


Fig. 3. Restrictions of mass balance per liberation classes.

The only residual higher than 6% refers to a tailings grade, with value of 0.13% copper, for which the absolute difference was of only 0.1 percentage point.

These results show that the plant was in steady state and was well represented by the samples, which allowed a good reconciliation with low residuals in all parameters. This global balance was appropriate for an evaluation per size-liberation classes.

4.2. Balance per size fraction

For each size fraction, the Cu, Fe, S and Si contents were transformed into concentrations of chalcopyrite (cpy), pyrite (py) minerals

and gangue. A new mass balance was obtained with these minerals per size fractions, setting the calculation parameters to make the balance per size compatible with the flows and concentrations obtained in the previous overall balance.

All flows presented correlation factors above 0.85 and the vast majority, factors above 0.9 between measured and reconciled values. Trendlines passing through the origin presented slopes very close to 1.0, being, in all cases, above 0.89.

Fig. 5 shows the correlation between measured data and reconciled values for all size fractions (+ 210 μm, - 210 μm + 150 μm, - 150 μm + 74 μm, - 74 μm + 44 μm, - 44 μm + 20 μm and - 20 μm) of all products (concentrate and tailings of rougher 1, rougher 2, scavenger,

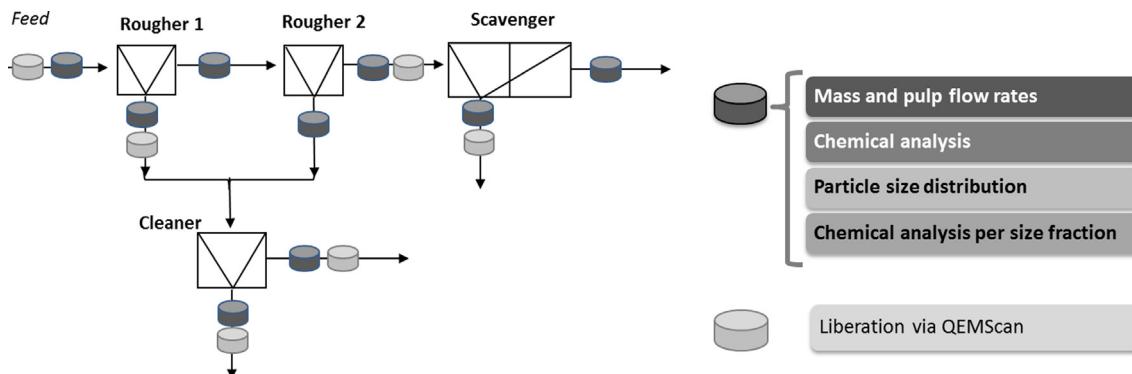


Fig. 4. Sampling points and analysis performed in each flow.

**Table 2**  
Sampling scheme.

Order	Product	Sampling time
1	Feed	15 s
2	Cleaner concentrate	2 h 30 min
	Cleaner tailings	2 h 30 min
	Scavenger concentrate	2 h 30 min
	Scavenger tailings	1 min aliquot at every 15 min during 2h30min
		5 min
3	Concentrate rougher 1 + 2	5 min
		2 h 30 min
4	Rougher 1 concentrate	2 h 30 min
	Rougher 2 concentrate	2 h 30 min
	Rougher 2 tailings	1 min aliquot at every 15 min during 2h30min
5	Rougher 1 tailings	1 min
6	Feed	1 min

cleaner, and feed).

The high correlation between measured and balanced data demonstrate that the size fraction reconciliation represents the process with good accuracy and is appropriate to be used as a basis for size-liberation balances

### 4.3. Balance per liberation classes

#### 4.3.1. Adjusting a beta function

For each size fraction of each product, a beta curve was adjusted to fit the liberation distribution.

Once data of all products had been adjusted by the beta curve, the whole set of liberation data from all test products could be reconciled.

At this point, it was necessary that the chalcopyrite grade calculated by the balanced data was equal to the grade obtained in the mass balance per size fraction. In a reconciliation software, this would mean assigning an error equal to zero to the size fraction grade, but this option of zero error precludes reconciliation. The use of Excel is a good option to perform this calculation.

Fig. 6 presents the distribution of measured data, beta distribution and mass balance for each particle size fraction of this same concentrate. Figs. 7 to 9 compare measured data, beta distribution values and balanced data.

Figs. 7 to 9 show that mass balance is compatible with adjusted beta curve, which in turn, is able to represent the measurements. Mass balance was successfully reconciled.

### 5. Effect of the changes in the approach

Mathematically, the original Savassi method was very well grounded. The removal of both gangue and liberated particles, as the original method recommended, seemed a very good artifice to the use

of incomplete beta curve to represent liberation. This approach worked well, but occasionally, gangue or liberated particles were over or underestimated in some fractions. This occurred because the parameter  $\gamma$  was used for removing and reinserting gangue and liberated particles, but the minimizing function was focused on the total grade and on the differences between the measured and adjusted composite particles. Then,  $\gamma$  was adjusted to reach the final grade, without considering the difference between the measured and adjusted proportions of liberated or gangue classes.

As mentioned, main changes were:

- In dealing with data
  - having the distribution expressed in 11 classes, incorporating liberated class to the precedent class. Then, liberated particles could be represented by the beta curve
- In the method
  - minimizing the sum of squares of gangue proportion as part of the minimization function
  - adding the weights in the minimization function

Mathematically, Fig. 10 illustrates the effect of these changes in the adjustment.

First plot (a) shows a beta curve that would be obtained using the original minimization formula, adjusting a distribution with 12 classes, for this particular fraction. In this specific case, liberated particles would be underestimated.

In a similar way, second plot (b) shows a beta curve that would be obtained using the original minimization formula without minimizing the difference of gangue proportion neither adding the factors, but adjusting a distribution with 11 classes, where fully liberated and almost liberated particles are a single class. In this case, gangue is overestimated.

The third plot (c) shows the beta curve adjusted with this optimized method, including the distribution with only 11 classes and the new minimizing function, including the difference of gangue proportion and the weight factors. Adjusted beta function obtained with this optimized method represented all composite classes, gangue and high grade particles.

### 6. Comments and conclusions

In a typical analysis of propagation error, there is a variable obtained by a mathematical function of measured parameters. The deviation in the unmeasured variable is calculated based on the errors in the measured parameters. In the case of a mass balance, it would mean that the size fraction mass flowrates would be obtained by the sum of liberation classes flow rates, and that global masses would be obtained by the sum of size fractions. In other words, in the regular approach, the

**Table 3**  
Global Mass Balance results – Test Cal 03.

	Solids flowrate		Pulp flow rate		Water		Copper grade		Chalcopyrite grade				
	Measured values (g/min)	Estimated values (g/min)	Residuals	Recovery	Measured values (g/min)	Estimated values (g/min)	Residuals	Estimated values (g/min)	Measured values (%)	Estimated values (%)	Residuals (%)	Recovery (%)	Estimated values (%)
Feed	114.0	111.2	2.4%	100.0%	345.6	358.0	3.6%	246.8	2.12	2.12	0.2%	100.0	6.1
Rg 1 Conc	8.5	8.7	2.4%	7.8%	20.9	20.6	1.4%	11.9	21.89	22.06	0.8%	81.6	63.8
Rg 1 Tails	104.3	102.5	1.7%	92.2%	339.8	337.4	0.7%	234.9	0.44	0.42	3.9%	18.4	1.2
Rg 2 Conc	2.4	2.4	0.1%	2.2%	19.8	19.3	2.3%	16.9	11.89	12.09	1.7%	12.4	34.9
Rg 2 Tails	104.2	100.1	3.9%	90.0%	325.6	318.1	2.3%	218.0	0.13	0.14	9.1%	6.0	0.4
Sc Conc	3.0	2.9	1.8%	2.6%	52.5	51.9	1.2%	48.9	2.29	2.27	0.8%	2.8	6.6
Sc Tails	100.9	97.2	3.6%	87.4%	282.6	266.2	5.8%	169.0	0.08	0.08	2.5%	3.2	0.2
Cl Conc	9.3	9.6	3.8%	8.7%	24.3	25.1	3.4%	15.5	21.38	21.27	0.5%	87.0	61.5
Cl Tails	1.4	1.5	3.1%	1.3%	14.1	14.8	5.2%	13.4	11.03	11.03	0.0%	7.0	31.9



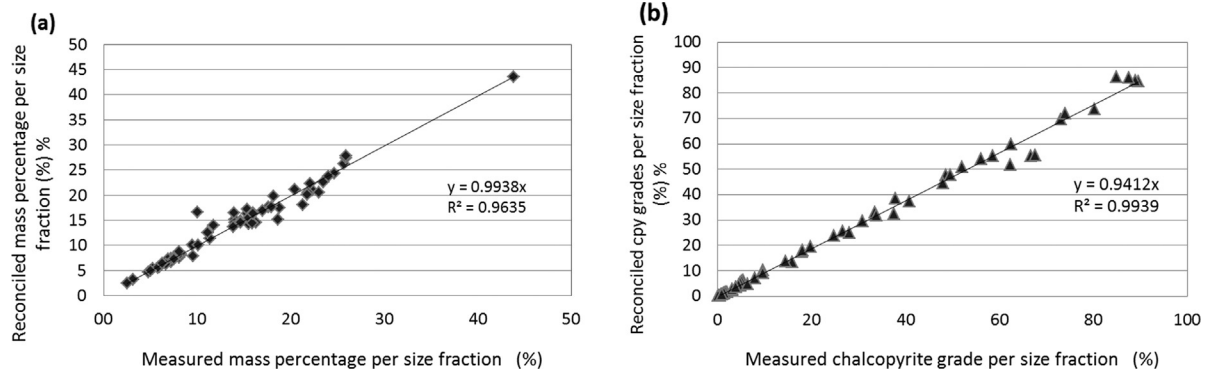


Fig. 5. Correlation between measured and reconciled values of size fraction mass balance of all products – (a) mass proportion (b) chalcopyrite grade.

“whole” is obtained by its parts. Since parts may vary significantly, the whole will vary much more.

Savassi method applies the reversal logic: it hierarchically

determines the domain of possible results, the “whole”, that will condition the subdomains or, its “parts”. Once the process testing and sampling are *physically* conducted to minimize errors, then the global

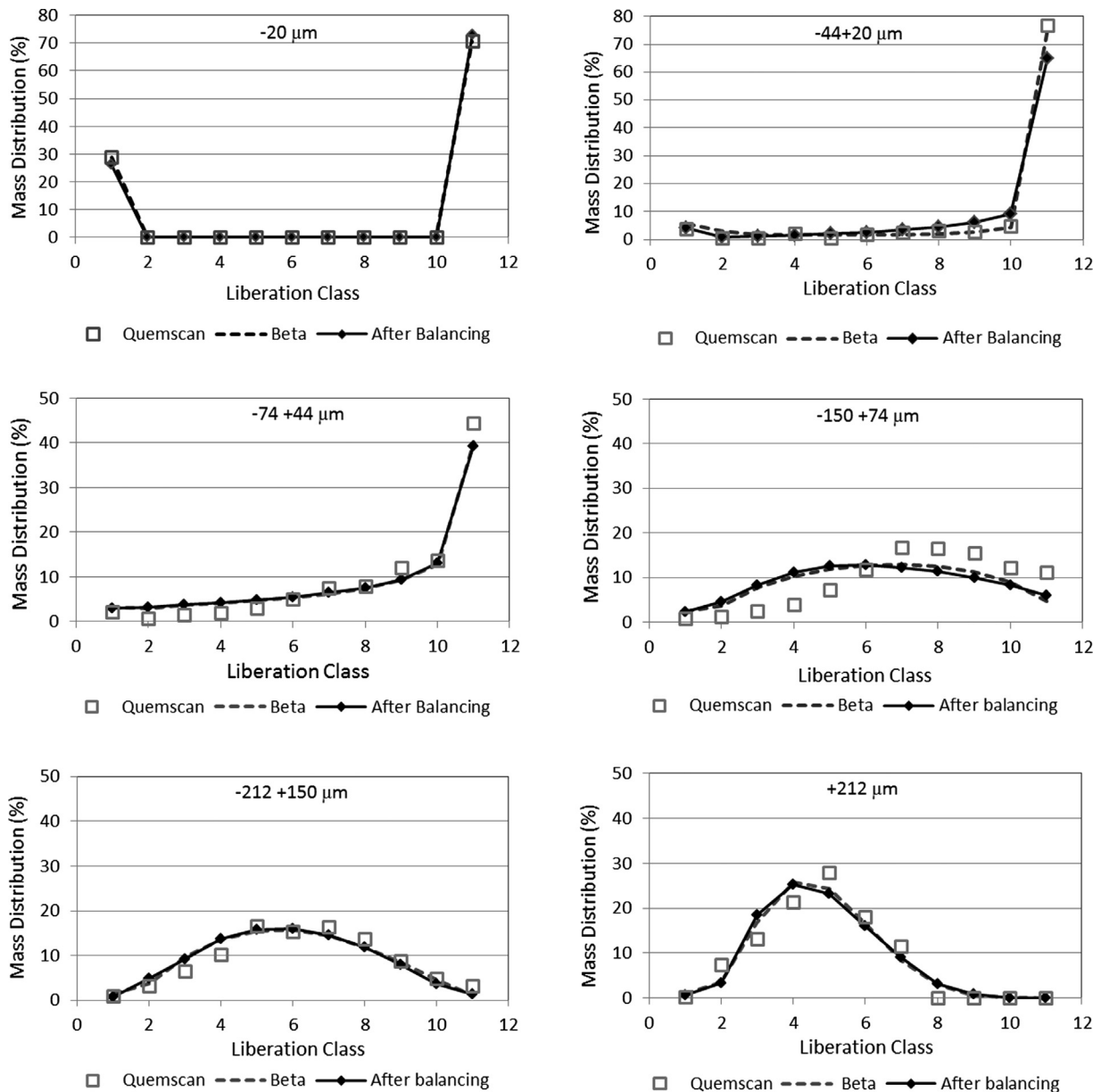


Fig. 6. Comparison between Qemscan measurements, beta function and balanced data for rougher 1 concentrate.

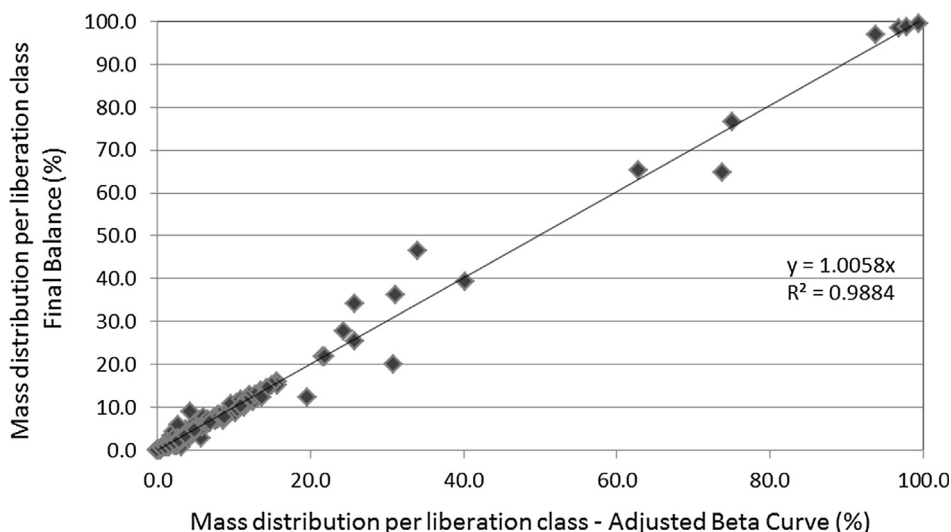


Fig. 7. Comparison between beta function and balanced data for all products.

error values are effectively lowered. Maintaining the global values at low deviations creates a restricted domain of possible values for the sublevels. This procedure increases the reliability of the calculated data of these sublevels. Or, since the whole has clear boundaries obtained by reliable global measurements, measured values of the parts are reconciled to fit the whole and respect all restrictions.

According to that, the conventional error propagation analysis does not apply to this mass balance reconciliation methodology.

Although the reconciliation per liberation class is an ill-posed problem with more than one solution, the data domain that attends all these restrictions is limited. In this sense, reconciled values per liberation classes are a set of data that is simultaneously compatible with:

- the measured data;
- all mass conservation restrictions among products within each level;
- values of previous and more reliable balances that better represent the process.

Thus, this mass balance has high probability of being representative of mineral liberation reality in the process. In other words, once this method explores the natural conservation laws in a hierarchical mass balance, the set of possible results is progressively restricted and

becomes very limited when reaches the level of liberation; therefore, most probably, the reached set of reconciled liberation data is representative, because it complied with all the interconnected restrictions.

The adjustments made in this work contributed to increase this representativeness. This reliability allows detailed process analyses which are the subject of other studies, where the behaviour of liberation classes in a continuous process was evaluated. (Santos, 2017; Santos and Galery, 2017).

As a suggestion for future developments, this method could be further improved to include multiminerall particles.

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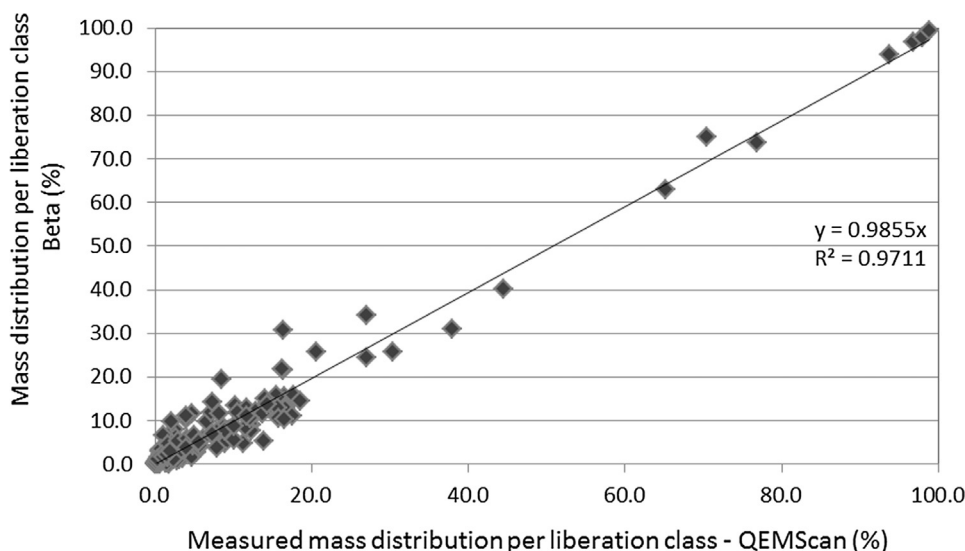


Fig. 8. Comparison between Qemscan measurements beta function for all products – Test Cal 03.

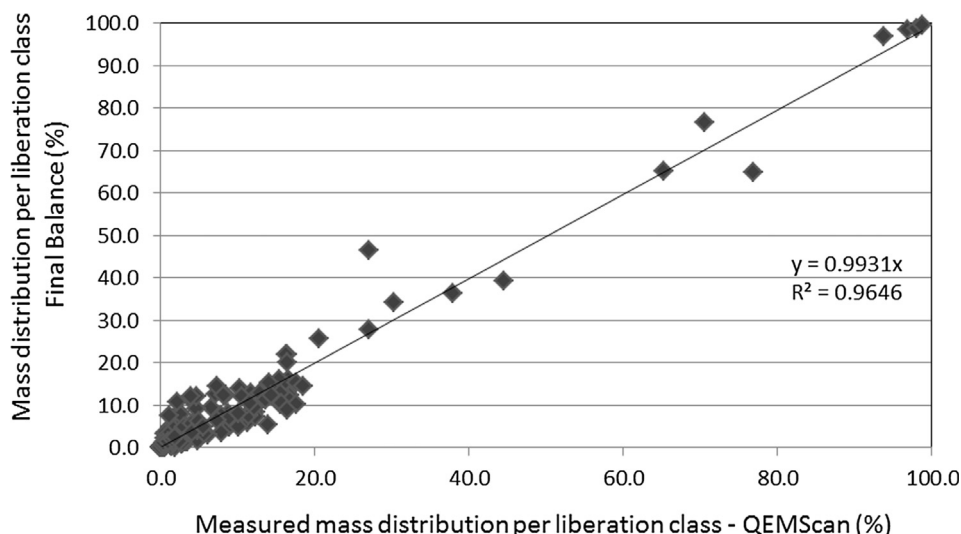


Fig. 9. Comparison between Qemscan measurements and final balance for all products – Test Cal 03.

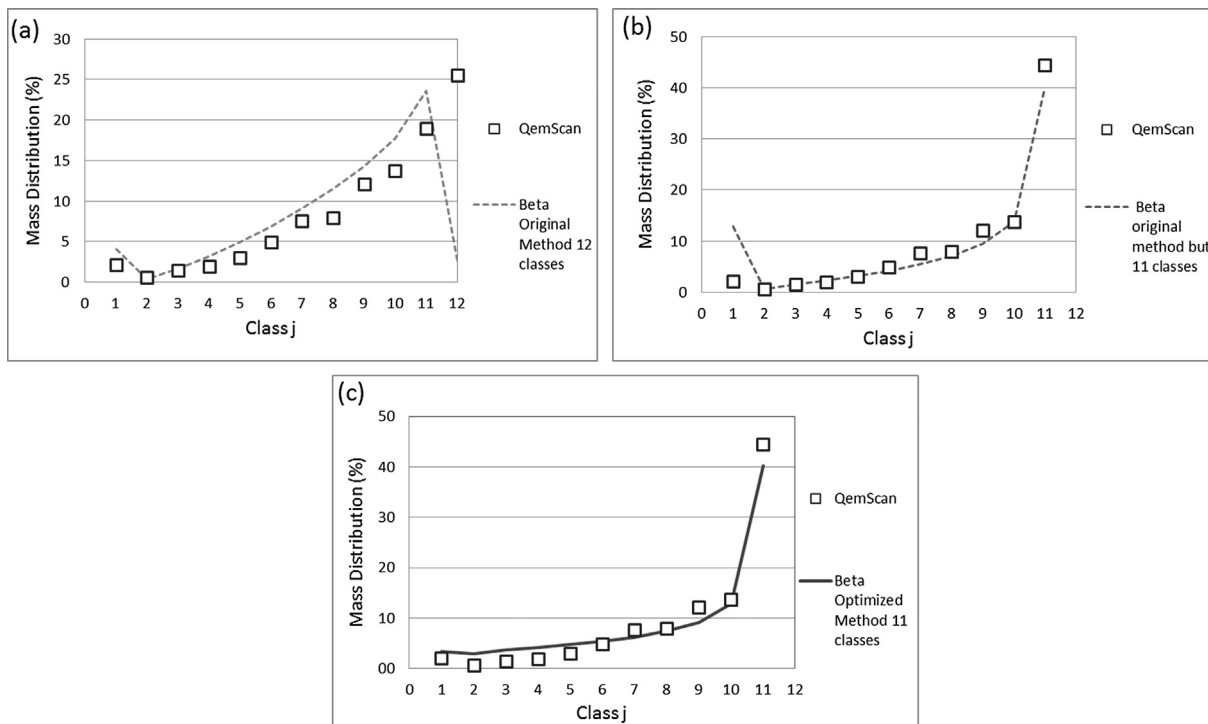


Fig. 10. Effect of changes in the method on the beta curve fit. Comparison between QEMScan measurements and beta fit for rougher concentrate, fraction –74 + 44 μm (a) with original method;(b) with original method but using only 11 classes; (c) with optimized method.

is acknowledged.

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