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Incorporating the covariance effect in modelling batch flotation kinetics

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ABSTRACT

In this paper, recovery covariances were used to estimate the kinetic parameters from batch flotation tests to account for the lack of statistical independence and homoscedasticity in the cumulative recoveries. Non-linear parameter estimations were compared by using unweighted least squares estimation (ULSE), weighted least squares estimation (WLSE) and non-linear generalized least squares estimation (NLGLSE). Three autocorrelated time-recovery curves were used as base case to simulate theoretical kinetic response. Single Rate Constant, Rectangular and Gamma models were employed to describe the kinetic response.

The NLGLSE allowed for significant precision improvements in the parameter estimation with respect to ULSE and WLSE, under known and constant covariance estimates. The variability of k_{SRC} (SRC), $k_{\max}$ (Rectangular) and k_{mean} (Gamma) decreased by approximately 40% with respect to ULSE, and 28% in comparison to WLSE. For R_∞, the dispersion decreased 33% in comparison to ULSE, and 17% regarding WLSE. The limitations of ULSE and WLSE were caused by a lack of validity of the assumptions of independence and homoscedasticity in the time-recovery curves. The advantages of NLGLSE were only observed with accurate estimators of the covariance matrix, which were obtained in the simulations and in a laboratory flotation test that involved 11 replicates.

Incorporating the covariance matrix in the parameter estimation allowed for improvements in the kinetic characterization. Thus, uncertainties related to the ore potential and circuit sizing (obtained from R_∞ and the rate constant estimates) might be decreased using accurate covariance estimators in the objective functions.

1. Introduction

Laboratory batch flotation tests are routinely performed in industrial operations to compare metallurgical results of different geometallurgical units as well as to define maximum achievable performances for continuous operation. In addition, kinetic characterization together with different scale-up methodologies have been also used to design industrial flotation circuits. For example, Dobby and Savassi (2005) suggested the use of the MinnovEX Flotation Test (MFT) to obtain standardized kinetic results. These results, along with simulations, have enabled the design of new and the reconfiguration of existing flotation circuits. Amelunxen and Amelunxen (2009a,b) reported a fast-pulling laboratory method to determine flotation kinetics at laboratory scale. The authors applied this model, supported by continuous pilot tests, to determine optimum configurations based on a cost-benefit approach. Boeree (2014) studied several variables such as turbulence, segregation, froth phase and machine dimensions, which differ significantly from laboratory-, pilot- and full-scale flotation systems. The generalization and applicability of the scaling-up methodologies were questioned based on the significant differences among all these systems.

Several model structures have been proposed to describe flotation kinetics at laboratory, pilot plant and industrial scale. Garcia-Zuñiga (1935) and subsequently Sutherland (1948) modelled flotation kinetics by means of a single first-order rate constant, similar to the approach for describing chemical reaction kinetics. Garcia-Zuñiga (1935) incorporated the maximum recovery, R_∞, to describe a final value that cannot be increased further. Sutherland (1948) also included an integral equation for mineral recovery, considering a distributed induction period for the valuable particles. A graphical method to describe the probability of flotation of two minerals was reported by Kelsall (1961), which discussed the effect of reagent dosage, fraction of locked minerals, particle size and type of flotation machine on the probability of flotation. The methodology, based on two rate constants and their relative content, was applied size-by-size at industrial scale on an ore consisting of chalcocite and malachite. Imaizumi and Inoue (1963) discussed the distributed nature of the rate constant in the flotation processes. As a result, a distribution function or spectrum was

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incorporated into the flotation rate equation. Woodburn and Loveday (1965) used a continuous Gamma distribution for describing this spectrum. The proposed probability density function (PDF) was successfully applied in batch flotation modelling as well as for predicting continuous performance of pyrite-silica separation. Klimpel (1980) determined whether the flotation process was controlled by the flotation rate or by the maximum recovery when reagents were changed. The author used a Rectangular PDF to describe the flotation kinetics at laboratory scale. The effect of changing reagent concentration on the kinetic response was employed to illustrate the methodology. Ferreira and Loveday (2000) proposed a flexible model consisting of the sum of two normal distributions. This distribution is defined by six parameters and numerical integration is required to obtain the predicted recoveries. The model fitting was tested with batch flotation tests of different streams from an industrial cleaning circuit. Dobby and Savassi (2005) used the k-distribution for modelling the batch flotation kinetics and for scaling-up the results to continuous operation. This PDF is similar to the Weibull, Whiten or other double-exponent functions. Other authors have proposed second order (Arbiter, 1951) or fractional calculus (Vinnett et al., 2015) approaches to describe flotation kinetics. However, these model structures have shown either equivalence with conventional first-order approaches or lack of robustness and physical significance (Alvarez-Silva et al., 2016; Bu et al., 2017; Dowling et al., 1985).

Goodness-of-fit of kinetic models have been extensively analysed and compared based on flotation tests at laboratory scale. Lynch et al. (1981) summarized a wide range of discrete and continuous distributions to describe the flotation rate. The authors pointed out that a better fitting is typically obtained as the number of parameters increases; however, the trade-off between the number of parameters and their physical significance must be considered. Dowling et al. (1985) evaluated thirteen model structures based not only on the goodness-of-fit but also on the confidence interval of the parameters. An extra parameterization caused the confidence interval of the parameters to become wider (dilution), where the models with a high number of parameters were typically reduced to simpler forms. Mazumdar (1994) compared five different model structures for the flotation rate constants based on statistical indexes related to goodness-of-fit and parameter stability. The results showed that no model was better than the others for the evaluated datasets. Polat and Chander (2000) reported that significant model fitting improvements are obtained when three-parameter models are used to describe flotation kinetics. This study also included sample fractioning by size and specific gravity. The flotation rate distributions approximated the classical first-order model for each size-by-specific gravity class. Bu et al. (2017) emphasised the overfitting phenomenon caused by the increase in the number of parameters in the kinetic models.

Bazin et al. (1995) studied the variability of laboratory flotation tests considering: (i) sampling and assaying errors; (ii) operator- and equipment-related errors; and (iii) the inherent random nature of the flotation process (*e.g.*, differences in liberation or collector adsorption of sub-samples). Based on experiments with different operators and an automated system, the authors concluded that the main source of variability was the random nature of the process, with lower contribution of the other two. Lotter (1995a,b) and Lotter and Fragomeni (2010) recognized the importance of accurate estimations of metal grades as well as the key role of subsampling, blending, replicating and monitoring to report reliable metallurgical indexes. A high confidence approach was proposed to decrease the uncertainties in the ore grade estimations, which is based on the Gy's minimum sample mass rule along with Safety Line models (Gy, 1979). In addition, Lotter (1995a) reported a quality control model that included the comparison between the sampled feed grade and total mass with those values calculated from the flotation products (concentrate and tailings). This model makes it possible reconciled and reproducible performance parameters to be obtained from laboratory flotation tests, under specific threshold values. Bazin et al. (1996) reported a data reconciliation methodology to evaluate error propagation in batch flotation indexes (recovery and cumulative grade). The data reconciliation results showed a slight variance improvement in terms of the metallurgical indexes in comparison to that obtained directly from measured data. The error propagation considered the variances and covariances among empirical grades and weights. Napier-Munn (2012) presented a complete statistical analysis to determine and compare grade-recovery curves from batch flotation tests. Empirical data from these tests were used to simulate ideal conditions for modelling and for comparison purposes. Although the auto-dependence among the samples was recognized, it was not included to relate the enrichment ratio with the mineral recovery. Sandoval-Zambrano and Montes-Atenas (2012) carried out an error propagation analysis for the flotation rate estimation based on the size-by-liberation data reported by Welsby et al. (2010), which consisted of flotation tests of a lead-zinc-silver ore in continuous operation. The effect of measurement errors on the flotation rate estimation was studied, assuming statistical independence among the input variables. Size-by-liberation recoveries as well as residence time showed the higher contributions to the rate constant variability, which presented coefficient of variation ranging from 11% to 121%.

Table 1
Summary of flotation reagents and dosages.

Identification	Commercial name	Chemical name	Dosage, g/t of ore
Primary collector	Matcol D-101	Modified dithionocarbamate	33
Secondary collector	Matcol SEC-301	Modified sodium di-isobuthyl dithiosulfate	7
Frother	F-749	Complex oxygenate/ hydrocarbon mixture	25

This paper presents the impact of using recovery covariances on the estimation of the kinetic parameters from batch flotation tests. From replicate tests, the sample covariance matrices were obtained. Such matrices were used as baselines to simulate kinetic data under different model structures (Single Rate Constant, Rectangular and Gamma). These simulations allowed opportunities for improvements in the kinetic characterization to be identified by including the covariance effect on the parameter estimation.

2. Flotation tests

Three samples of a porphyry copper ore deposit were used for flotation tests. These three samples have chalcopyrite ($CuFeS_2$) as the main copper phase within a silicate matrix. The ore samples were crushed using firstly a laboratory jaw-crusher and secondly a roll crusher to obtain a particle distribution 100% passing 10 Tyler mesh (1.7 mm). The sample was blended, riffled and then split using a 6-bottle rotary splitter with a vibratory cone hopper to ensure homogenization. The subsamples were used for: (i) a grinding test; (ii, iii, iv) three flotation tests at different operating conditions (no kinetics); (iv) ore grade determination; (v) flotation kinetics; and (vi) left as witness sample. Further riffling (by adjusting an appropriate bottle module to the rotary splitter) was required to obtain 750 g-subsamples used in grinding and flotation tests. Minor mass adjustment to the subsamples were required thereafter. Thus, batches (iv) and (v) were used in this study. The same procedure was successively carried out according to the ore availability, for the three different samples.

The grinding time to obtain a P_{80} of 150 µm was previously determined for the three ore samples to be 27.0 min, 30.2 and 25.2 min, respectively. The solid was placed in a ball mill with 750 mL of distilled water and lime was added as a primary pH control. Similar to the grinding time, a prior study was conducted to adjust the pH of the pulp product at approximately 10. Thus, 295 g/ton, 185 g/ton and 220 g/ton