

## **AMINPRO'S METHODOLOGY FOR EXECUTING AND INTERPRETING LABORATORY BATCH KINETICS TESTS (PART 1 OF 2)**

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### **ABSTRACT**

For many years traditional laboratory flotation tests have been used, in combination with empirical scale-up factors, for establishing the flotation residence time and metal recoveries of commercial flotation circuits. Recently, research has led to a better understanding of the contribution of the froth zone to the overall flotation cell performance, allowing design engineers to replace the old empirical methods with modern phenomenological ones. As a result, the scale-up procedures have become based more on science and economics rather than individual experience and benchmarking exercises. The authors' approach tailors the test procedure to provide data in a form that allows the test interpreter to discriminate between collection zone recovery and froth zone and entrainment effects, thereby producing collection zone kinetic parameters that can effectively be used for scale-up. This paper provides describes the approach and provides a historical context for the methodology.

**KEYWORDS:** laboratory flotation; phenomenological models; scientific scale-up.

## 1. INTRODUCTION

The batch flotation kinetic test has become the standard means for establishing the residence time and mineral recoveries of commercial flotation circuits. The output of a traditional flotation kinetic test is a curve of recovery vs. time, such as that shown in Figure 1. It is the process engineer’s job to “translate” the kinetic curves provided by laboratory batch test work into flotation circuits, or simulations of a flotation circuit, composed an arrangement of multiple, industrial scale, continuously fed flotation cells.

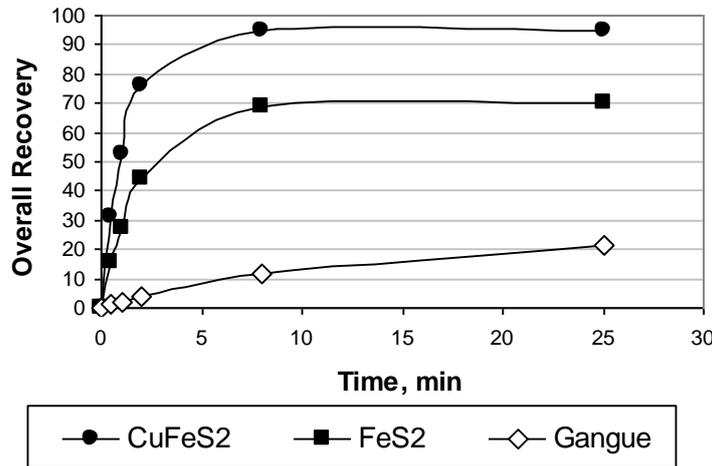


Figure 1: Laboratory Kinetic Test Output.

The history of flotation modeling is broad, deep, and interesting in its own right. A comprehensive historical review is provided elsewhere by one of the authors (Amelunxen & Runge, 2013) and will only be summarized here.

Since at least 1935 it was suspected that batch flotation followed first order kinetics (Garcia-Zuñiga, 1935). For a plug flow reactor, the mineral recovery can be described as a function of time using the well known first order rate equation (after Morris, 1952):

$$R_c = (1 - e^{-kt})R_\infty \quad (1)$$

Where  $R_c$  is the recovery by collection at time  $t$ ,  $k$  is the first order rate constant [ $\text{min}^{-1}$ ] and  $R_\infty$  is the projected recovery at infinite time. Throughout the ensuing decades, many studies were conducted to refine the concept. It is impossible to summarize all the studies here, but many of them sought to relate the rate constant,  $k$ , to various process phenomena such as:

- Attachment and detachment rates (Schuhmann 1942),
- Ore characteristics such as particle size (Gaudin *et al.* 1942, Morris 1952)
- Mineralogy and particle oxidation (Imaizumi and Inoue 1963),
- Operating conditions such as reagent dosage or reagent surface coverage (Schuhmann, 1942), and
- Machine-dependent characteristics such as bubble saturation or impeller energy input (Sutherland, 1948).

During the 1950s test procedures focused on determining the kinetics mainly for those minerals that would generate money for the project; little was done for those, such as pyrite and gangue, which provided no income. Furthermore, an accepted method for scaling up the results from a laboratory kinetic test to commercial level did not yet exist. Many scale-up methods appeared, of which the use of flotation time factors became the most widely accepted. For example, Lindgren and Broman

(1976) believed that the plant flotation time should be estimated by applying a factor of two to the laboratory time at the point where the recovery levelled off.

One drawback to this method was that to correctly determine the value of the multiplier, extensive personal experience was required with the particular flotation process in question, and this experience was often marketed as a competitive advantage (albeit a somewhat unverifiable one) by the various researchers or equipment manufacturers. Another drawback relates to the fact that because each lab technician might “pull” the froth at his or her characteristic speed, reproducibility of the tests, particularly between labs, was often poor.

It is of interest that this method is still being applied today, in a great number of projects, with safety factors added (as they were in the 1950s) to account for tonnage fluctuations, grade fluctuation, contingency and so on. In reviewing flotation history, some early advances in the field of chemical reactor engineering deserve mention. One of the earliest calculations of flow and kinetics in continuously fed stirred tank reactors (CSTRs) is provided by Ham and Coe (1918). In 1935—the same year that Garcia-Zuñiga proposed the exponential equation for modeling batch test recovery—the first discussion of short-circuiting in CSTR cells was published (MacMullin and Weber 1935). It wasn't until the 1950s, however, that the modern discipline of chemical reactor design began to take shape. P.V. Danckwert's comprehensive analysis of residence time distributions was perhaps the most influential technical contribution (Danckwerts 1953). The field was still in its infancy, however, and the First European Symposium on Chemical Reaction Engineering, held in Amsterdam in 1957, provided the cross-pollination of ideas necessary for the discipline to crystallize. This conference was ultimately credited by Octave Levenspiel as the inspiration for his “Chemical Reaction Engineering,” which was published in 1962 and was to become one of the most cited works in chemical engineering history (Levenspiel 1962, Current Contents 1979). And so it was that one of the earliest continuous mixer models appeared in extractive metallurgy 1962. It was derived from first principles by Arbiter and Harris and represented as:

$$R_c = k\tau / (1 + k\tau) \quad (2)$$

Where  $\tau$  is the reactor mean residence time. It should be noted that the first application of Equation 2 to extractive metallurgy is generally credited to Arbiter & Harris (1962), but it also appeared in a flotation text by Dorenfeld (1962). It is significant, because it marks a clear departure from empirical or heuristic scale-up methods used earlier, and although its adoption was neither immediate nor widespread, it clearly formed the basis of all phenomenological scale-up approaches that were to come.

It was a milestone in froth flotation modeling, as most of the subsequent developments were small, “evolutionary” changes to this same approach and the table title, no lines between the title and the table and two single lines between the table and the subsequent text. The previously described Arbiter & Harris (1962) publication was also exceptional because it was among the first to:

- Note that Equation 2 is only valid for the collection zone,
- Suggest that high confidence kinetic data could only be achieved by rapid froth removal from the laboratory cell to avoid “particle drop-out from the froth,”
- Propose a kinetic model based on particle size (they even lamented that computer technology was not sufficiently advanced to handle the computationally intensive algorithms that would result).

Around the same time, it was recognized that the heterogeneous nature of the ore feeding a flotation cell gives rise to differences in floatability between the particles (Imaizumi and Inoue 1963). Researchers proposed alternatives to the first order rate equations for plug-flow reactors (PFRs) and

CSTRs, depending on the kind of model being proposed. Because the exact shape of the rate constant distribution could not be accurately measured, methods were proposed to approximate the shape by fitting the curve parameters to experimental data. Runge (2007) classifies the approach according to the method used to approximate the shape of the distribution. These are:

- Empirically-derived distributions, in which the shape of the distribution is defined by a small number of parameters that can be derived from experimental data. Examples include the rectangular distribution (e.g. Klimpel 1980), the triangular distribution (e.g. Harris & Chakravarti 1970), and the normal distribution (e.g. Chander & Polat, 1994).
- Empirically-derived flotation rate components, in which the feed particles are grouped into discrete classes and the mass and flotation rate of each class are fitted to experimental data. The best example is the Kelsall model (1961).
- Property-based rate components, in which the feed is discretized into classes based on physical characteristics such as size class and mineral type. The flotation rate for each size class is then fitted independently to the experimental data. Examples of this approach have been provided by Thorne *et al.* (1976) and Kawatra *et al.* (1982).

The goodness of fit of the laboratory kinetic test curve using Eq. 1 has not always been satisfactory. Agar *et al.* (1980) suggested that a better fit to the curve would be achieved if Garcia-Zuñiga's equation had a time adjustment,  $\Phi$ , for the test as shown in Equation 3 (below). The adjustment was justified for the elapsed time between when the air is turned on and the first froth is removed into the pan.

$$R_c = \left(1 - e^{-k(t+\phi)}\right) R_\infty \quad (3)$$

## 2. AN OVERVIEW OF CURRENT FLOTATION MODELS

With the advent of column flotation in the early 1980s, research funds were made available to study column flotation. Models began to evolve to describe the column cell froth zone, and these were later applied flotation cells in general. Lynch (1981) associated the recovery of hydrophilic particles with water recovery to the concentrate. Warren (1983) improved the entrainment concept by applying entrainment to hydrophobic particles as well.

The first compartmental models began to emerge, such as the approach of Finch and Dobby (1990) described by the familiar depiction shown in Figure 1. The fractional recovery by flotation,  $R_{flot}$ , for the system in Figure 1 may be defined as:

$$R_{flot} = \frac{R_c R_f}{R_c R_f + 1 - R_c} \quad (4)$$

Where  $R_c$  is the collection zone recovery and  $R_f$  is the froth zone recovery.

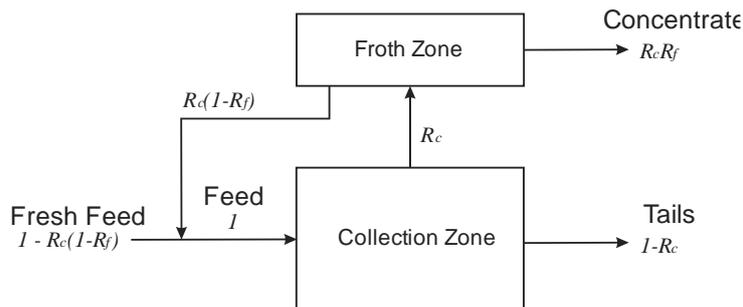


Figure 2. Mass Balance of a Flotation Cell Incorporating Froth Zone.

Froth recoveries were rarely applied in laboratory test work interpretation, despite the warnings of Arbiter and Harris. Although some researches found little selectivity in froth recoveries (Vera, *et al.*, 1999), others (i.e. Honaker, 2005) reported that weakly floatable mineral species had preferential detachment in the froth zone.

In the late 1990s and early 2000s, research into the froth zone behavior intensified. It had been recognized since at least 1972 that the rate of entrainment of particles into the concentrate is linearly proportional to the water recovery (Johnson *et al.* 1972), yielding the well-known equation for degree of entrainment (D.E.), shown here in the form published by Savassi (2005):

$$D.E. = \frac{R_s}{R_w} \quad (5)$$

Where  $R_s$  is the solids recovered to the concentrate by entrainment and  $R_w$  is the water recovery to the concentrate. The recovery of particles by entrainment was first incorporated directly into the first order CSTR model recovery model by Savassi (1998). The equation is now used by most plant simulators, and it is written as follows:

$$R_{tot} = \frac{k\tau(1 - R_w) + ENTR_w}{(1 + k\tau)(1 - R_w) + ENTR_w} \quad (6)$$

For batch tests, Equation 6 is rearranged and the term  $kt/(1+kt)$  is replaced by the plug-flow reactor model. Savassi (1998) made many measurements of the degree of entrainment and suggested three types of relationships: a high degree of entrainment (Curve A) with preferential drainage of coarse particles, an intermediate curve (B) for preferential drainage of coarse particles and curve C representing a low degree of entrainment. All three models predict that slime-sized particles will entrain into the concentrate at approximately the same rate as water.

The incorporation of a froth zone model and entrainment effects into the interpretation of laboratory batch flotation processes was suggested by Vera *et al.* (2002). Dobby *et al.* (2002) reported the incorporation of entrainment recovery but made no mention of a froth zone recovery model for rate constant determination.

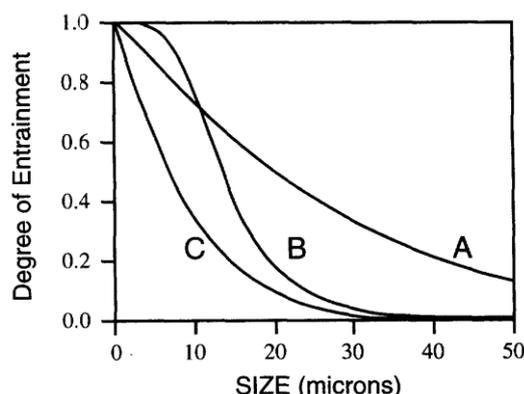


Figure 3. Entrainment curves per Savassi (1998).

In parallel with the advancements in froth zone modeling, research into the fundamentals of the collection zone model were also progressing. Polat *et al.* (2000) expanded the concept of rate constant distribution by relating it to particle size. Dobby and Savassi (2005) proposed working with more abstract cumulative distribution functions, termed  $k$ -distributions, that are, at least initially, determined through particle size distribution measurements in the lab. Gorain *et al.* (1998) proposed a relationship between the bubble surface area flux through the collection zone ( $S_b$ ) and the first order collection zone rate constant; Deglon *et al.* (1998) further developed the idea.

Researchers began to express the rate constant as a linear function of  $S_b$ . There is some discussion on the precise nature of the relationship (for example, see Heiskanen 2000), but one such expression, proposed by Hernandez *et al.* (2003), relates the rate constant to the values of  $S_b$  and froth recovery:

$$k = PS_b R_f \quad (7)$$

Where P is the “floatability factor,” which encompasses the particle size and hydrophobicity. Given the findings summarized by Equation 7, it should not be surprising that in recent years there has emerged an extensive body of research aimed at predicting the bubble surface area flux as a function of various chemical, mechanical, and metallurgical parameters.

To close the historical discussion, the authors’ approach to flotation circuit modeling is summarized as follows, in the context of the previous discussion:

1. When entrainment and froth recovery effects (see below) have been included in a proper mass balance and interpretation methodology, the standard first order kinetic equation fits the laboratory batch test data extremely well. Klimpel rate constants can NOT be used with the classical first order perfect mixer model (Equation 2).
2. The authors agree with Arbiter and Harris (1962) that the froth should be removed as fast as possible to minimize particle drop back and disagree with the assumption of 100% froth recovery in the lab, as implied by Dobby *et al.* (2002) and observed in practice at many laboratories. Laboratory froth recoveries are lower, sometimes significantly so, for some slow-floating mineral species.
3. The authors agree with Agar and have adopted Equation 3 for batch test kinetic modeling.
4. The incorporation of a froth zone model, complete with froth recoveries and entrainment effects, is of paramount importance for accurate laboratory measurement of kinetics parameters.
5. The authors prefer to work directly with the kinetic rate constant rather than  $S_b$  and “floatability factors”. Given the current debate on the nature of Equation 7, combined with the challenges of predicting the plant scale bubble surface area flux at the project design stage, the authors suggest that prior to test work, a laboratory cell calibration procedure be performed such that gas rate, impeller RPM, and frother type are optimized to give the highest rate constant. This calibration procedure is analogous to the optimization procedure that occurs in full scale flotation circuits after commissioning.

The authors prefer to use the rate constant vs. particle size curve directly, rather than the more abstract frequency distributions proposed by Dobby and Savassi (2005). This approach facilitates the modeling of regrind and cleaner circuits by tracking each size class independently without the need to recalculate the distribution parameters every time a grind size is changed.

### 3. AMINPRO’S BATCH PROCESS INTERPRETATION MODEL

#### 3.1. Test Procedures

Aminpro has two kinetic test procedures depending on user needs. These are shown in Figure . To obtain a single kinetic value of  $R_\infty$  and  $k$  for each mineral, the SKT test procedure (“Simple Kinetic Test”) is used. For a distribution of kinetic values as a function of particle size the FKT (“Full Kinetic Test”) procedure is used.

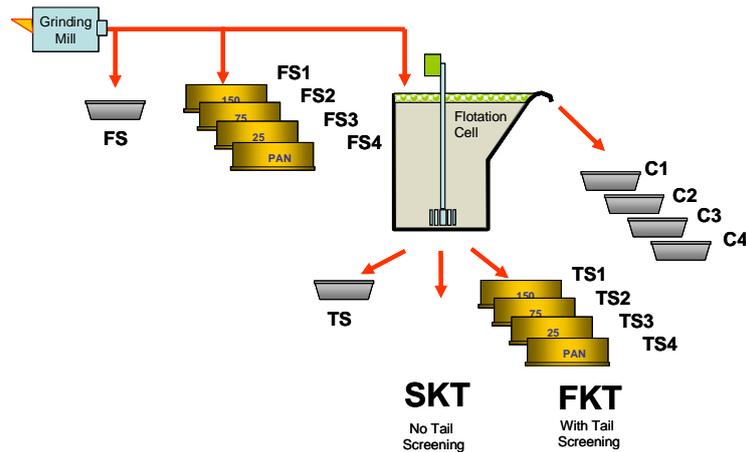


Figure 4. FKT and SKT Test Procedure Flow Diagram.

The FKT test requires screen analysis of the feed and the tails and each size fraction is assayed. The SKT only requires a size analysis of the feed and no assays of the fractions are required. The feed sample is initially ground to a desired  $P_{80}$  passing size. The product from the mill is placed in the flotation cell where a measured volume of feed is extracted for head assay and screen analysis (mass balance calculations consider the feed mass after removal of the feed subsample). Reagents and modifiers are added to the lab cell and are conditioned for 1 minute and the air is then turned on and the clock is started. As soon as the mineralized froth appears, the operator starts scraping the concentrate into a pan at a rate of approximately one scrape per second. The fast scraping rate is necessary to maximize froth recovery and prevent drop back of hydrophobic materials into the pulp zone.

Concentrate is collected in four intervals, timed to yield approximately the same weights. For roughing the intervals often are 0-  $\frac{3}{4}$  minutes,  $\frac{3}{4}$  to 2, 2 to 8, and 8 to 30. The test is run continuously, without stopping between intervals. Water is added during the test as needed to maintain cell level, and staged reagent addition is permitted in order to better reflect stage addition in the full scale circuit. At the end of the test all products are weighed as slurry and as a dry product prior to assaying.

### 3.2. Data Reconciliation and Mass Balancing

To minimize the influence of the sampling and assay error, both FKT and SKT tests are designed to provide assay redundancy to correct the assays and close the mass balance. This can be done by using commercially available tools or with Microsoft Excel's solver algorithm. Size distributions obtained from the screen analysis are represented by the Swebrec function (Ouchterlony, 2005).

### 3.3. Laboratory Flotation Model

The overall recovery is determined through the interaction of three processes: a collection zone recovery process, a froth zone recovery process, and an entrainment recovery process. These sub models may be represented in a simplified manner in Figure 2 where  $R_{flot}$  is defined by equation 4. From the equations for  $R_c$  (Equation 3),  $R_{flot}$  (Equation 4), and D.E. (Equation 5), the total recovery can be expressed as follows:

$$R_{tot} = R_{flot} + (1 - R_{flot})R_e \quad (8)$$

Where  $R_e$  is the recovery by entrainment and is equal to  $R_s$  in equation 5. The interpretation model seeks to replicate the recovery-time curves of all minerals floated in the test by using Equation 8.

This is accomplished by fitting values of  $R_{\infty}$  and  $k$  for each mineral while simultaneously solving for  $\Phi$ ,  $R_f$  and  $R_s$  for each time interval during the flotation test.

#### 4. COMPARISON OF THE TRADITIONAL AND AMINPRO APPROACH

A series of FKT tests were conducted on a copper/moly ore in a commercial lab in Chile to examine the differences in kinetics between the Aminpro method and the lab's traditional procedure (slow scraping rate). The tests were performed with identical starting conditions and flotation times. The resulting recovery vs. time curves, shown in Figure 5, clearly indicate that for both copper and moly the faster scraping rate yields significantly higher mineral recoveries and flotation rates. It also results in a higher mass recovery and a correspondingly lower concentrate grade. Note that because the test is designed to measure the collection zone kinetics of each mineral, for modeling purposes, the average concentrate grade of the batch test is irrelevant. This point may be counter-intuitive, so it is worth repeating: *the average concentrate grade from the batch test is irrelevant for the purpose of modeling recoveries and concentrate grades of a full-scale flotation circuit.*

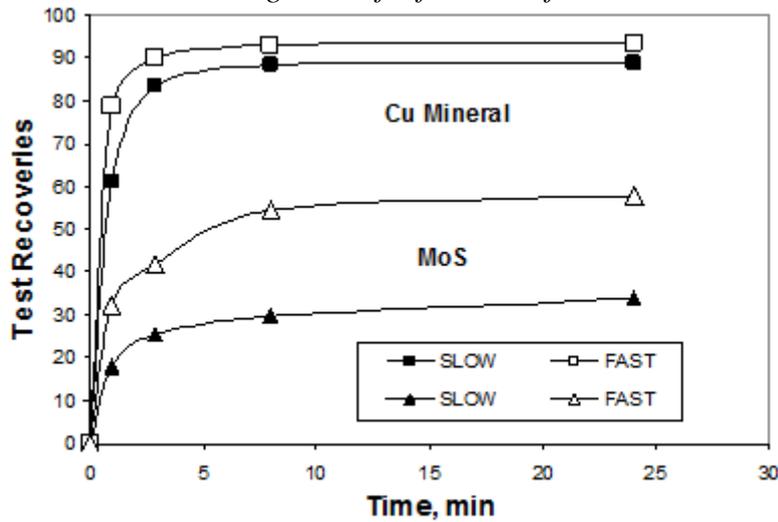


Figure 5. Comparison of kinetics for different pulls under identical starting conditions.

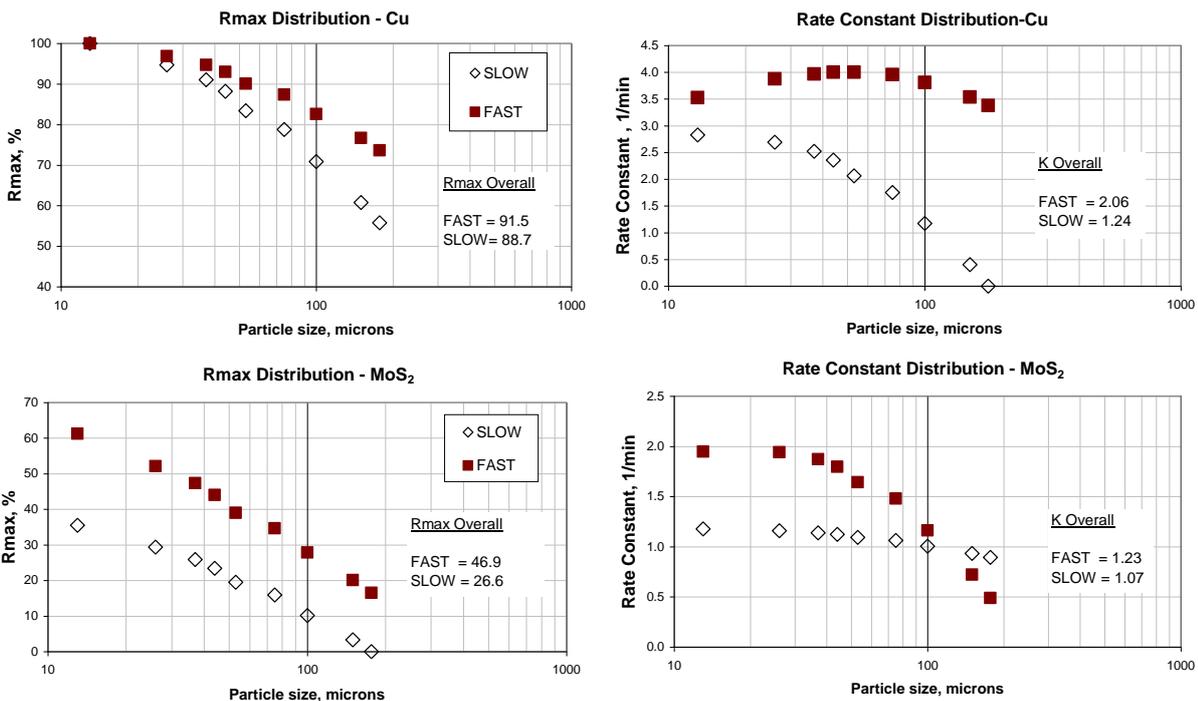


Figure 6. Collection zone kinetics of copper sulfides and molybdenite as a function of particle size.

The resulting  $R_{\infty}$  values for copper and moly are significantly higher with the fast pull, by approximately 2.8% for copper and 20.3% for molybdenum. The difference in  $R_{\infty}$  can be explained by examining the kinetics as a function of particle size determining with the FKT test. In the FKT test interpretation, the minerals' kinetic parameters,  $R_{\infty,i}$  and  $k_i$  values, are determined by setting the sum of the screen recoveries,  $R_{c,i}$ , equal to the overall collection zone recovery for each time interval:

$$R_C = \sum_{i=1}^n R_{c,i} = \sum_{i=1}^n (1 - e^{-k_i t}) R_{\infty,i} X_i \quad (9)$$

Where  $X_i$  is the mineral distribution in the  $i$ th size fraction. The results, plotted in Figure , show  $R_{\infty}$  and  $k$  as functions of particle size. It is immediately apparent that the copper losses in the slow-pulling test are occurring primarily in the coarser size fractions, while the moly losses are occurring throughout the entire size range with significantly lower rate constants in the fines.

In this example, the process plant designed using the Aminpro kinetics would likely yield, on paper, higher copper and moly recoveries and a significantly different economic optimum cleaner circuit regrind size. The example serves to illustrate the authors' precept that for a plant design based on kinetics and mathematical modeling, *kinetic data must describe, as closely as possible, the true physicochemical reaction of the unit operation being scaled*. Because flotation is a compartmental process, even at the batch scale, the reaction rates are best measured by applying procedures that allow for timely reaction measurements without promoting ambiguity between the "compartments".

## 5. CONCLUSIONS

This paper has presented an overview of Aminpro's batch flotation test procedures and interpretation methodology in the context of historical modeling developments. A methodology has been provided for extracting collection zone kinetic parameters after accounting for froth zone effects and mechanical entrainment, and a comparison was provided showing that the Aminpro methodology has the capacity to yield significantly higher, and more realistic, mineral flotation kinetics than those resulting from traditional methodologies. The increased flotation rates and recoveries allow the process engineer to design flotation circuits that would yield significantly higher mineral recoveries than those derived from traditional bath test methods.

The collection zone kinetic parameters determined using the new methodology are directly applicable to plant scale flotation cells. The resulting collection zone recovery is subsequently built upon by adding back the froth zone and entrainment effects, corrected for the plant scale froth depths and lip distances. This will be the subject of part two of this series.

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