Introduction

Flotation is a concentrating process that has been around since 1860 with the first patent by William Hayes. Early in the 20th century flotation evolved into the process familiar to metallurgists today. The drive has always been towards optimizing the grade and recovery profile of the flotation process.

A plant survey enables the collection of relevant data over a process. The collected data allows an understanding of the process at the time of the survey and can be used for modelling. Software packages are available to assist in the mass balancing and modelling with simulation options to predict an outcome to process changes.

The value of the collected data to the specific process is, however, very labour intensive and have long turnaround times. Error is introduced in the form of contamination, sampling methods, and sample preparation techniques, and by the time the survey data is available and mass balanced, the process conditions may have changed significantly. Having a rapid in-line measurement of grade enables the metallurgist to take steps to optimize the recovery in the main stream flotation banks whilst ensuring that the required grade in the final concentrate stream is maintained. The technology used for the in-line measurements is based on diffuse reflective spectroscopy together with advanced chemometric methods. The precision and accuracy of this technology as well as its considerations towards safety and robustness is described. Data is updated several times per minute, which comfortably allows feedback control to manipulate air rates, reagent dosages or pulp levels towards a grade or recovery set point. In addition, the measurement of grade can be used as a tool to evaluate the effect of changes to the manipulative variables on PGM grade and Cr₂O₃ impurity levels when attempting process improvement.

Process stabilization

It is commonly known that stabilization precedes optimization. Stability can refer to minimized deviation between a parameter’s set point (SP) and process variable (PV) or to minimized deviations in a parameter over time due to process change. In this context, stability will refer to the latter.

During a flotation survey, care is taken to ensure that the feed flow rate and density is stable (not fluctuating at a high frequency). Reagent addition flow rates are checked where flow meters are available and reagent spot checks are done on older plants. Operators are strictly requested to not make any changes to flotation pulp levels, air rates or any other parameters for the duration of the survey. The stability is monitored throughout the survey and the survey is terminated as soon as a mill trip or other process upset occurs. The dynamic process is now assumed sufficiently stable for the survey.

What happens in the background? Given ‘stable conditions’ it is fair to assume that variation in the percentage solids, product grade, and impurity level in the produced concentrate per cell is minimal and attributed only to uncontrollable process factors. It has, however, been realized over the years that flotation performance stability is not as trivial as merely ensuring constant reagent addition, air rates, and pulp levels. The flotation process is much more dynamic than generally accepted.

Data were collected for a four hour period from a single rougher concentrate on a platinum concentrator to evaluate the response of percentage solids and PGM (platinum group metal) grade to air addition.

Figure 1 illustrates the dynamics of the percentage solids and their air variation in the cell. Within a range of 1 m³/min, the variation in percentage solids was 10% absolute. The relative standard deviation (RSD) of the percentage solids over the four-hour period was 14%.
The PGM grade in Figure 2 illustrates a significant variation (RSD) of 7% over a range of 1 m³/min. For an air addition of 9 m³/min, the PGM grade ranged between 230 and 310 g/t.

The question that arises is whether the air addition to a cell should be kept constant or the PGM grade and recovery be kept stable. A stable air rate to a cell does not secure a stable PGM grade nor constant entrainment of Cr₂O₃. This is also true for stable reagent addition or fixed pulp levels. If a constant air addition to a cell delivers a varied grade, mass pull and recovery, the process is not really stable during a survey.

Plant survey: the bucket method

Plant surveys are mostly done using ample amounts of plastic buckets and a team coordinated to take samples at planned intervals for a predetermined period dependent on the residence time of flow through the circuit of interest.

Manual samples are taken with pelican beak and long-handle sample cutters. Volumetric flow rates are measured using a bucket with known volume and a stopwatch where flow rates are low and sump-rise tests for higher flows. Densities are measured using an electronic densimeter or a Marcy scale. A sampling team is considered fortunate if some of the sampling points have auto samplers that can be operated in manual mode and more so where flow rates and densities are available from installed instruments.

Sampling campaigns are also done to collect hydrodynamic data from flotation cells and also to collect samples for lab re-floats. In many cases the data are extremely valuable for modelling and simulation of a circuit and also to calculate floatabilities.

The downside of sampling surveys is that it does not capture the true dynamics of the process and that the data turnaround time limits its value to the process. In short, it does have value, but the value decreases with every passing minute after the survey has been concluded without the results being available.

Other risks with manual samples are incorrect sampling, incorrect sample handling, weighing and splitting errors, and finally analytical errors.

Why survey data become outdated

Operators and metallurgists throughout the industry have different ideas on what the best ‘recipe’ is for a successful operation. Some plants have a set range of air rates and pulp levels that is adjusted as required to accommodate process changes, instrument drift, and temperature fluctuations. There are plants that prefer not to change air rates but rather pulp levels or reagent dosages.

During the 3–4 hours a plant survey is done, these parameters are not changed. If the same survey is repeated during the night, the value of the parameter chosen by the operator or metallurgist will most probably be different from that chosen during the day as generally more air is introduced into the cell during cooler air conditions. If the survey is repeated a month thereafter, the picture will look entirely different.

Plant maintenance is generally done once a month during a planned maintenance shutdown. An example of maintenance that affects operating parameters is mill relining which affects the fineness of the grind and particle size distribution to the flotation feed.

The replacement of worn flotation cell rotors and stators improves the flow of air through a cell. The same amount of air introduced into the cell before will therefore produce a higher mass pull afterwards.

Artisans routinely recalibrate instrumentation and it happens from time to time that mistakes are made. An example is the measuring range between the ultrasonic level instrument and plate attached to the buoy floating on the pulp interface. If the measuring range should be 1 metre from the lip into the cell and this is wrongly calibrated to 0.8 metre, operating at a pulp level set point of 80% will not be 20 cm from the lip, but 16 cm.

Reagent tanks are washed and lines are flushed out during shutdowns. This is especially important if organic guar depressants are used. If the depressant becomes mouldy it can affect the flotation performance.

All the above can affect the operating points of the air rate, pulp level, and reagent addition. These factors should be taken into account and best would be to repeat a survey after a shutdown to ensure relevancy.

In-line grade measurement: real-time data

In a typical sulphide flotation circuit in the platinum industry, real-time grade data have many valuable applications.

Flotation feed

The % Cr₂O₃ in the primary flotation feed can be measured for ore quality control and to make adjustments to reagent addition if required.

The throughput is dependent on the availability of ore. The availability of continuous grade data can assist in evaluating the options of either running at a fixed throughput until the silos are almost empty or reducing
throughput to avoid an unnecessary shutdown. Generally a lower throughput positively affects the recovery, but decreases the final concentrate grade significantly.

Dependent on the availability of mineralogical analysis for calibrations, mineralogy data can be made available in realtime. The data can be used for pH corrections by controlling the addition of a pH modifier to ensure an optimal environment for the flotation of the applicable ore type the PGMs are associated with (pyrite, chalcopyrite or pentlandite).

Flotation performance
The PGM grade and % Cr$_2$O$_3$ in flotation concentrates can be controlled by adjusting parameters such as air rate, pulp levels or reagent additions. Recovery can also be controlled if mass flow rates are available on the feed to the flotation bank and the concentrate line where the grade is measured. Even though the grade of the feed to the bank is also required for a recovery calculation, incremental PGM grade changes in mainstream flotation feed are relatively insignificant.

Process control has many advantages; but in-line grade measurements can also be used for flotation test work. A matrix of air rates, pulp levels, and reagent dosages can be evaluated without hard labour. On-off plant trials can be done to compare different reagent types or evaluate stage dosing or any other process option.

When it is decided to switch over from one steel ball type to another, it can take months before the effect can be evaluated as the ball load in the mill is slowly replaced. Real-time data are of value to do a comparison of this nature. The effect of relining is, however, immediate and a change should be seen between the real-time grade data before and after the shutdown.

Troubleshooting
Real-time grade data are valuable to identify equipment malfunction. A typical example is a sliming float cell due to dart valve or vent captor malfunction.

Cost saving
The entrainment of Cr$_2$O$_3$ is a major concern in platinum concentrators treating UG2 ore. Some concentrators incur penalties by the smelters treating their product. There is a significant financial benefit in controlling these impurity levels to specification.

In-line grade measurement for process control
Instruments used for process control in the flotation circuit have in the past been described as sparse and unreliable and the control challenged by the varying dynamics and interaction between process units.

Multi-stream elemental on-line X-ray fluorescence (XRF) analysers became available on the market to measure samples that have been extracted from process streams. An advantage is that multiple streams can be measured at once, enabling comparison between feed, concentrate and tails. Composite samples can also be analysed quickly for metal accounting purposes. A disadvantage is that samples need to be extracted representatively from the stream prior to measurement. Secondly, the higher the number of streams measured, the further apart the intervals of data and the less significant the data become for process control. The measuring time per stream is typically adjustable between 15 and 60 seconds with an analysis cycle estimated at 1 minute per stream. Therefore, for every additional stream measured, the interval between grade measurements increases by the stream’s measurement duration.

A technique using reflectance spectroscopy has been developed and is available for in-line grade measurements. Grade and % solids data was captured over 140 minutes at intervals of 15 seconds (0.25 minutes) to estimate the required measurement interval for sound process control.

PGM grade data sampled at different intervals
The dynamics of the PGM grade is illustrated in Figure 4 to Figure 7. Figure 4 concludes that a measurement interval of 1 minute is sufficient for process control as most peaks and variation in data are captured.

PGM grade data captured inline every 5 minutes as depicted in Figure 5 starts showing signs of aliasing. Most peaks are not visible and suggest a less significant change to the PGM grade than what is evident from data captured every 15 seconds.

Figure 6 suggests that intervals of 10 minutes are disregarding not only most of the peaks in the data but also oscillations in their entirety. At this point it becomes clear that manipulative action by a controller based on data captured every 10 minutes cannot be accurate.

Capturing grade data at 20 minute intervals from composite samples is of value for metal accounting. It is, however, clear from Figure 7 that measuring the grade of a stream every 20 minutes has no value for process control. The controller will chase only a ghost image of the data as aliasing takes over.
Diffuse reflectance spectroscopy

Reflectance spectroscopy studies reflected and scattered light as a function of wavelength and has many applications in the food, agricultural, pharmaceutical and minerals processing industries. Diffuse reflectance occurs as incident light is reflected into all directions from the surface because of scattering. Light not absorbed or transmitted by the surface material (solids, liquids, and gas) make their way to the detector (spectrometer).

The reflected light is a function of amongst other things particle size and the vibrational characteristics of all the molecules and crystals in the surface material being scanned.

As a large number of factors affects the shape of the obtained spectra, propriety chemometric methods have been developed to calibrate the optical spectra to many characteristic (grade, % solids, etc.) changes that result in a spectral (complex colour) change.

The instruments operate in the ultraviolet, visible and near infrared (VNIR) spectral range (wavelengths of 350–1 100 nm).

The technique is inherently safe as no nuclear radiation or X-ray sources are involved. In addition, the technique is robust for use in the harsh plant environment. Technical maintenance is concluded with a light source change once a year, leaving only monthly calibration as an ongoing consideration. The optical data is calibrated against analytical data obtained from X-ray fluorescence and fire assays (PGMs).

Accuracy and precision

The accuracy of the data is highly dependent on the accuracy of the calibration data. This is best illustrated by Figure 8 and Figure 9 for the PGM grade. Process step changes were done and calibration samples were collected every 10 minutes. The PGM grade responded well to the changes but had an offset, especially at the lower range, as it has not been calibrated on such low values previously.

The downside of the technology is its dependence on the quality of the calibration data. The accuracy of the calibration sample assays is paramount to the accuracy of the measurements.

After the new calibration data was included into the calibration set, the predicted PGM grade values improved significantly.

The precision of the data is very good as homogenous material is scanned in bulk and not only a small prepared non-homogenous sample.

The correlation between the assay and measured data was already very high ($R^2$ of 96%) before the update of the
calibration even with the offset, which confirms that even with an offset in data, the measurements are still relevant for process control.

After the calibration, the correlation improved to an $R^2$ of 98%.

**Conclusion**

The stability of a process can be validated only with the availability of frequent data. Real-time grade data suggests that a stable air addition does not result in a stable PGM grade or % solids in the concentrate.

Frequent data are not only valuable for process control, but also as a diagnostic tool for process upsets, for data analysis, quick turnaround times for test work, and process comparisons.

Sampling surveys are valuable for academic and modelling purposes, but their value to the process itself is limited. Surveys are labour intensive, error prone, and have long turnaround times. The impact of ore changes are often recognized, but that of plant maintenance is overlooked. A flotation process is very dynamic, necessitating frequent measurement to make the correct changes to air addition, pulp levels, and air rate set points for process optimization.

Data at intervals of one minute apart are highly relevant for process control. At longer intervals, aliasing occurs and the value of the data diminishes rapidly.

**References**


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