Sulfide Flotation Testing

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Laboratory batch flotation testing is a fundamental part of characterizing a sulfide ore to determine the merits of recovering valuable minerals from an ore. This chapter describes the laboratory procedures for characterizing the flotation response of sulfide ores and products from sulfide flotation plants.

LABORATORY TESTING FOR SULFIDE FLOTATION AND PROCESS DEVELOPMENT

A carefully planned laboratory test program is essential for the successful development of any sulfide flotation process. These programs usually begin with a series of batch tests on several ore-type composites to evaluate appropriate reagent schemes and delineate optimum rougher flotation conditions, such as primary grind size, flotation pulp density, flotation pulp pH, and retention time. Cleaner parameters such as regrind size requirements, cleaner pH, pulp density, and number of stages are then defined. Semicontinuous locked-cycle tests are performed using the parameters established in the batch tests to determine the effect of middling streams recirculation on flotation performance. The data generated and the experience gained in the locked-cycle tests may be used to design and

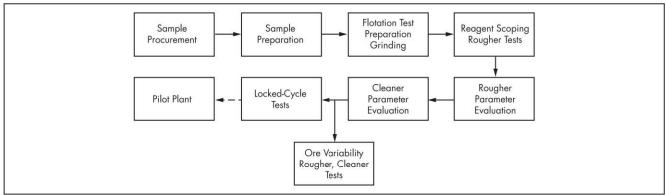
operate a continuous pilot plant if relatively large amounts of concentrate are required for further process development.

A comprehensive ore variability program is undertaken to determine the response of various ore types to the optimum conditions established. These ore variability programs often test hundreds of samples in standardized batch rougher and cleaner flotation tests to ensure that the ore body is well understood (Thompson 2016).

The importance of obtaining representative samples, along with careful preparation and preservation, is essential to all metallurgical test-work programs. The most careful and comprehensive test programs are meaningless if the samples used are not representative. The block flow diagram in Figure 1 provides an overview of the development process.

Batch Laboratory Flotation Equipment

Several standard laboratory flotation machines are available. The most common are the Denver D12 (supplied by Metso) and the FLSmidth-Essa. The machines have impeller speed control, and air is controlled by a rotameter. Air pressure is typically set at $\sim 50-75$ kPa.



Source: Thompson 2016

Figure 1 Generalized flow diagram for sulfide flotation development

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Batch Laboratory Flotation Test Cell Sizes

The most common flotation test cell sizes used in sulfide flotation work range from 1.5 to 6 L and typically contain 500–2,000 g of ground solids. However, flotation cells as small as 1.25 L and as large as 60 L are available. The larger cells are normally employed when considerable amounts of concentrate are required for additional testing, such as selective flotation to recover by-products (i.e., molybdenite), leaching, or gravity separation. The smaller flotation cells, 1.25 and 1.5 L in size, are generally used for concentrate cleaning applications. The 2.2-L and 4.4-L Agitair cells are typically used in many laboratory flotation test-work programs. Sample masses are typically 1 kg for the 2.2-L cell and 2 kg for the 4.4-L cell.

Batch Flotation Operating Parameters

Pulp Density

Choosing an appropriate flotation pulp density is often dictated by the gangue constituents of the ore, particularly the clay or slimes content. These types of slimes can have a significant effect on pulp viscosity, particularly if a high lime pulp pH is required for pyrite rejection. Typical pulp densities range from 30% to 40% solids in roughing and 15% to 20% solids in cleaning. The higher pulp densities are normally employed when treating siliceous ores with very low slimes content.

Flotation Reagent Conditioning

Suggested impeller speed for reagent conditioning is typically between 750 and 950 rpm for a Denver batch flotation machine using a 2.5-L batch cell. For low-intensity conditioning, the impeller speed range can be decreased to 600–750 rpm, whereas for high-intensity conditioning, increased impeller speeds to 900–1,250 rpm are typical.

Rougher-Scavenger Flotation

Recommended operating conditions provided are to be used as guidelines and are not "cast in stone." Cell revolutions per minute are chosen, usually on a visual basis, to provide adequate agitation and suspension of solids but should not be too intense to cause spillage. The intensity of agitation reduces once air is introduced into the system. Typical impeller speeds for rougher-scavenger flotation range from 950 to 1,500 rpm, depending on the mixing characteristics of the ore. Aeration rates can range from 5 to 25 L/m and largely depend on the froth characteristics of the sample being tested.

Cleaner-Recleaner Flotation

Typical impeller speeds for cleaner-recleaner flotation range from 700 to 900 rpm, depending on the mixing characteristics of the ore. For concentrates with large quantities of coarse particles, the impeller speed may need to be increased to 800–1,100 rpm to keep particles in suspension. Aeration rates can range from 0.1 to 5 L/m and, again, depend largely on the froth characteristics of the concentrate being floated.

Pulp and Froth Levels

With no agitation and airflow, the pulp level is set at 50 mm below the froth overflow lip. The cell should be marked and calibrated for pulp level with agitation and no air so that with airflow, the pulp level is 15–20 mm below the froth overflow lip. Froth height should be level with the overflow lip. During the rougher test, water needs to be manually added to the flotation cell to maintain the "set" pulp level. For the scavenger

part of flotation, the pulp level is usually increased by 5 mm below the froth lip. Depending on the pulp and froth characteristics of the ore being tested, the pulp levels will be altered by the operator (EMC 2012a).

Froth Scraping in Batch Flotation

Froth removal is usually by manual scraping by hand paddles, though mechanical scrapers can be incorporated into the system. The froth paddles are sized to extend 10 mm below the overflow lip (EMC 2012b). The typical scraping rate is one scrape (both sides of the cell) every 10–15 seconds. To increase the mass (yield) of concentrate, the scraping rate can be increased to 6–10 per minute.

Flotation Time

Rougher-scavenger flotation is typically carried out for 6–12 minutes. However, for ores where the sulfides are slow floating, the flotation time is extended to 20 minutes. For cleaning applications, floatation times are typically 70% of roughing times. Exceptions can occur, particularly when clayey ores are processed, where flotation times can be greater than the rougher retention time.

SCOPING FLOTATION TESTS FOR REAGENT SCHEME SELECTION

The first phase of most flotation test programs is the selection of a reagent scheme. This should be done in comparative rougher scoping tests before other important parameters, such as grind size, pulp pH, and pulp density, are optimized. A good starting point for sulfide systems is a primary grind P₈₀ of ~106 μm, pulp pH of 8-9, and pulp density of 30% solids. Flotation reagents may be classified into the following categories: collectors, depressants, activators, frothers, and pulp dispersants. Pulp dispersants include sodium silicate, for slimey or clayey pulps, and activators include copper sulfate for pyrite activation and for sphalerite flotation. Both flotation systems are not discussed in this chapter but can be found elsewhere in the handbook. Frother evaluation in small bench-scale tests is not effective because of the small surface area in these machines. Most reagent scoping tests are therefore focused on the selection of appropriate collectors (Thompson 2002), which may be separated into the following classes:

- Xanthates: Powerful, nonselective, minimal frothing, and relatively inexpensive.
- **Dithiophosphates:** Moderately selective, with some frothing characteristics.
- Thionocarbamates: Selective, with some frothing characteristics.
- Aerophines: Very selective, with moderate frothing characteristics.
- Mercaptobenzothiazole: Selective, specialty reagent for tarnished sulfides.
- Dual-action collectors: Reagents tailored for dual mineral recovery (e.g., copper and gold, copper and molybdenum).

In sulfide flotation systems, pyrite is the most common sulfide gangue mineral, and most depressants are evaluated for rejection of this mineral. Notable exceptions include the use of zinc sulfate for sphalerite depression in galena flotation and sulfur dioxide gas to depress galena in copper sulfide flotation. The most common pyrite depressants are inorganic

compounds such as lime (CaO), which is the most common depressant used; sulfites such as sodium sulfite, sodium metabisulfite, and sulfur dioxide gas; and sodium cyanide, which is the most powerful pyrite depressant but is seldom used because of environmental and social concerns. Several organic pyrite depressants are available, including calcium lignosulfonate, diethylenetriamine, sodium thioglycolate, 2-naphthelenesulfonic acid, modified polyacrylamide, and several starch/sodium hydroxide/sodium sulfide formulations.

For auriferous pyrite and arsenopyrire flotation, the standard collector is potassium xanthate in combination with a strong frother. To increase free gold recovery, if present, and improve overall performance, other flotation reagents are usually employed, as discussed by Kappes et al. (2011) and Dunne (2016).

The selection of a suitable reagent scheme is often made using an experience-based approach and begins with a review of existing operations that are treating ores with similar characteristics. Assistance from reagent vendors is recommended. Most screening flotation tests are performed in rougher kinetic format with at least three, and preferably four or five, timed concentrate samples taken.

ROUGHER FLOTATION PARAMETER EVALUATION

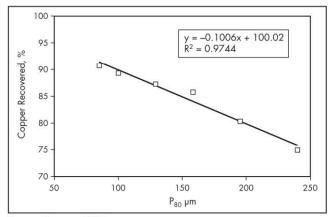
Rougher flotation parameters are normally evaluated in batch rougher flotation tests that are performed in a kinetic format after a suitable collector reagent scheme has been selected. These parameters include primary grind fineness, flotation time (kinetics), flotation pulp density, and flotation pulp pH. Primary grind fineness is the parameter that has the greatest impact on capital and operating expenses and, as such, is tested extensively. Rougher flotation testing focuses on valuable mineral recovery, but some selectivity against pyrite is often desired, and this is usually achieved by increasing the grinding and flotation pulp pH with lime. It is important to add lime to the grinding step prior to flotation to achieve maximum pyrite depression using this reagent.

Primary Grind Particle Size

The effect of the primary grind size of the primary grind on valuable mineral recovery is evaluated in a series of separate rougher kinetic tests at increasing grind fineness. Grind size P_{80} values varying from 75 to 250 μ m are commonly used, but coarser and finer grinds are occasionally tested. Figure 2 shows a typical plot for recovery versus grind size for a copper sulfide ore.

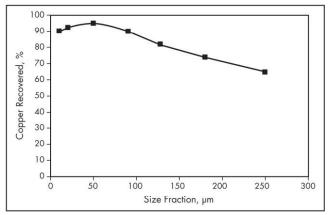
The negative slope of the linear regression analysis indicates that recovery decreases as the grind size becomes coarser, which is from locking of valuable mineral, usually with non-sulfide gangue. Slopes ranging from -0.1200 to -0.0245 are common for copper-molybdenum porphyry ores.

Size-by-size analyses of the flotation feed, the overall concentrate, and the tailings at a single grind size are often performed to provide a size-by-size recovery profile and to confirm the results obtained in the grind variability study. In these tests, each size fraction of flotation concentrate and tailings is weighed and assayed to calculate recovery by size fraction. The size-by-size analysis of the feed is used as a check on the metallurgical balance. Typical results are presented in Figure 3. This size-by-size test is used in most ore variability studies.



Source: Thompson 2016

Figure 2 Typical plot for copper recovery versus grind fineness



Source: Thompson 2016

Figure 3 Size-by-size recovery of copper for a typical ore

Occasionally, a more detailed size-by-size test is performed to evaluate both recovery and kinetics by size fraction. This type of test usually requires a 10- to 15-kg test charge because all the timed concentrate samples are screened, weighed, and assayed separately. The data obtained are usually worth the extra time, effort, and samples required. A schematic for this test and typical size-by-size kinetic curves are illustrated in Figures 4 and 5.

Size fractions >20 μ m (635 Tyler mesh) are generated using standard wet screen sieving. Size fractions finer than 20 μ m may be generated using a Marc Technologies Cyclosizer (formerly known as the Warman Cyclosizer). This device uses a series of either five or seven small, inverted cyclones in series to generate closely sized subsieve, or smaller than 20 μ m, fractions. The actual size distributions of each cyclosizer product may be determined using an appropriate laser particle size analyzer. The size-by-size kinetics data presented in Figure 5 indicate much slower kinetics in the coarser size fractions. This is typical for many ores because of the partial locking of valuable mineral with non-sulfide gangue-inhibiting kinetics.

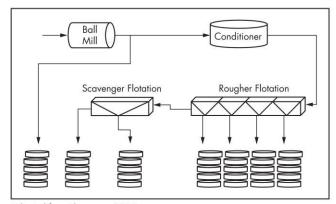
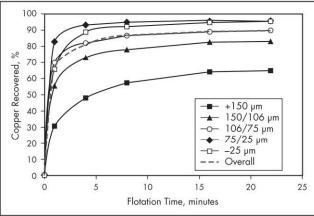


Figure 4 Flow scheme for size-by-size kinetics



Adapted from Thompson 2016

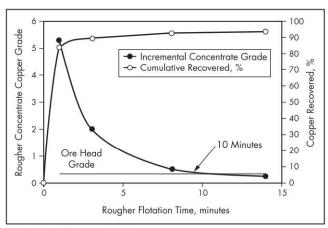
Figure 5 Size-by-size kinetics for a typical copper ore

Flotation Kinetics

Batch flotation tests are usually performed in kinetic format by taking flotation concentrates at several time increments until the froth is barren. The data are then presented in a kinetic graph similar to the one in Figure 5, using overall recovery because size-by-size kinetics are a special case.

The most widely used method for determining retention time in these batch tests is the cumulative recovery—incremental concentrate grade approach. Cumulative recovery and incremental grade of the individual time interval concentrates are plotted as a function of total flotation time. Flotation is considered complete when the incremental concentrate grade crosses the test feed head grade, drawn as a horizontal line. At this point, no additional concentration from the test feed material is occurring, and the test can be considered complete. Figure 6 illustrates this concept, which is valid for the evaluation of rougher, scavenger, and cleaner flotation.

Notably, flotation kinetics are generally influenced by two important factors, namely, the rate constant and the froth-carrying capacity. The second factor is often overlooked, but it can be an important one, particularly in cleaner circuits. Maximum froth-carrying capacity for industrial mechanical cells is provided in Chapter 7.4, "Froth Management," and froth-carrying capacity for column cells is discussed in Chapter 7.2, "Column Flotation." Typical values for sulfide ores tested in mechanical laboratory cells are 0.05 to 0.10 t/h/m²



Adapted from Thompson 2016

Figure 6 Rougher retention time evaluation by incremental concentrate grade

for roughing applications and 0.15 to 0.30 t/h/m² for cleaning applications. Kinetic limitations due to froth-carrying capacity in laboratory tests are generally limited to industrial minerals applications where high concentrate weight pulls are common. Column cleaning in some sulfide systems can also encounter this issue, particularly if the ore is high grade.

Flotation Pulp pH

In most sulfide flotation systems, the choice of flotation pulp pH is governed by the amount of pyrite rejection that is required. Ores containing high levels of pyrite often require rougher pulp pH levels of up to 12 for pyrite control, whereas ores containing more moderate or low amounts of pyrite may only require pH 8. Lime is the most common pH modifier used in the minerals industry. A series of rougher flotation tests at increasing pH values is recommended to determine the response of both pyrite and valuable mineral to this variable. Some collectors do not perform well at elevated pH values above 9. The sensitivity of cleaner flotation to pulp pH should also be investigated.

CLEANER CIRCUIT EVALUATION

Test programs for evaluating cleaner circuits should be undertaken only after rougher parameters are optimized. Although rougher testing focuses on maximizing recovery of valuable mineral, cleaner testing centers on producing a saleable concentrate of acceptable grade. In most sulfide systems, this is accomplished by separating the valuable mineral from nonsulfide gangue and sulfide gangue, usually pyrite. A delicate balance exists between grade and recovery in these tests, and the production of the highest possible grade concentrate may not always be the most economically viable, given the reduced recovery issues. Consequently, the most important product from cleaner flotation work is the grade—recovery curve.

Methods for Generating Grade–Recovery Curves

The standard approach for generating grade—recovery curves is a relatively simple method that is the most widely used in the minerals industry for most cleaning applications. Concentrates are refloated, and tailings from each stage are analyzed separately. Intermediate concentrates are back-calculated from

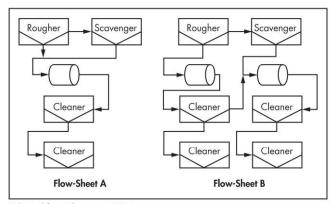


Figure 7 Two cleaner circuit configurations for comparison of grade–recovery curves

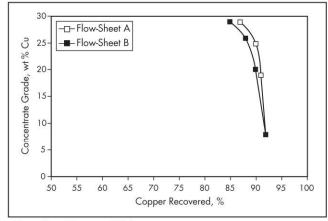
concentrates and tailings. Two other methods (release analysis and three circuits) are discussed by Thompson (2016).

Cleaner Circuit Configurations

Several large copper-molybdenum-porphyry concentrators clean rougher and scavenger concentrates in separate cleaner circuits. These types of separate cleaning circuits are exceedingly difficult to test in the laboratory because of factors such as low weight pulls in the scavenger-cleaner section and middlings recirculation from the rougher-cleaner to scavenger-cleaner sections. Figure 7 shows a simple and relatively complex cleaning circuit (flow-sheet A and flow-sheet B, respectively), while Figure 8 provides the laboratory graderecovery results for the two circuits. The curves are very similar, indicating that the relatively simple flow scheme A is acceptable for generation of laboratory grade-recovery data in most cases, and this example is used in the following sections that address cleaner circuit parameter optimization. These parameters include regrind fineness, flotation kinetics, number of cleaning stages required, circuit pH, and circuit pulp density.

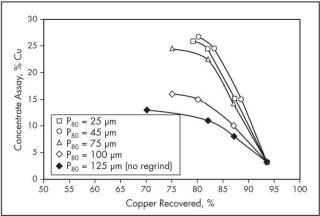
Evaluation of Regrind Fineness

Most rougher and rougher-scavenger concentrates that are produced in the rougher evaluation stage of a test program contain locked particles of valuable mineral and either non-sulfide gangue, often silicates, or sulfide gangue, usually pyrite, or both. Regrind testing is performed to determine the grind fineness that is required to produce an acceptable final concentrate grade. This test work is usually complemented with liberation and locking analyses by an automated mineral analyzer program. Pyrite may also be present as liberated mineral may report to the rougher concentrate because of activation by collector, entrainment in the froth, or both. In many copper sulfide flotation circuits, rougher flotation is considered a "bulk float," and pyrite flotation is tolerated to maximize copper recovery. This liberated pyrite must also be rejected during cleaning. Lime is the most common depressant used in these cleaner circuits, and a high lime cleaner pH is normally used when testing regrind fineness for copper ores. However, auxiliary depressants such as sulfides or cyanide may be required, particularly when treating ores where the valuable mineral is sensitive to calcium ion, such as lead-zinc-silver ores.



Adapted from Thompson 2016

Figure 8 Grade–recovery curves for two laboratory cleaner circuit configurations



Adapted from Thompson 2016

Figure 9 Effect of regrind fineness on grade-recovery curves

A high-lime cleaner pH of 11.0–11.5 is normally used in the concentrate regrind test series for copper ores. The recommended procedure consists of performing a large-scale rougher test on 12–15 kg of ore under optimum conditions. The rougher concentrate is then split into four or five samples using a rotary slurry splitter or equivalent. Each rougher concentrate split is then subjected to regrinding for increasing time periods, then cleaned at least two times, often more, at pH 11.0-11.5. The rougher concentrate is screened at an appropriate size, usually 25 or 37 µm, and only the screen oversize is reground. This prevents excessive sliming of the cleaner feed. This procedure generates a family of grade-recovery curves similar to those in Figure 9 for a copper-porphyry ore. Steep grade–recovery curves indicate good upgrading, and the steeper the curve, the more selective the cleaning. Evaluation of these results suggests that a regrind fineness P₈₀ of 45 µm is appropriate for this sample.

Number of Cleaning Stages

The shape of the grade-recovery curve and the desired final concentrate usually dictate the number of cleaner flotation stages required. Figure 10 shows two examples of different

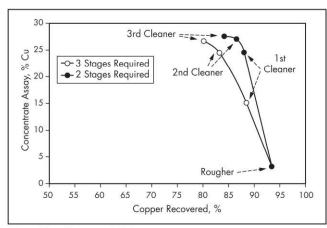


Figure 10 Typical grade-recovery curves for two- and threestage cleaning

cleaning requirements. Horizontal sections of the curve typically indicate no additional upgrading, and the cleaner stage at which the knee in the curve occurs may be considered the final stage. In some cases, the curve intersects the desired final concentrate grade before the knee in the curve—as in Figure 9 for the 45- and 25-µm regrinds if a 25% copper grade was targeted—and often there is no knee in the curve and the desired final concentrate grade may not be achieved. This is the case in Figure 9 for the P₈₀ of 100-µm regrind and 125-µm (no regrind) conditions.

The evaluation of cleaner circuits in the laboratory with mechanical test cells can be complicated if column cells are being considered for the full-scale plant. Column flotation testing is difficult in the laboratory given the large amounts of flotation feed slurry required, particularly in cleaner flotation. It has been author Thompson's experience that approximately three stages of mechanical cleaning in the laboratory approximates one stage of column cleaning in the plant.

Most cleaner circuits include a scavenging step on the first cleaner tailings, and this is always the case when column cleaning is used. The cleaner-scavenger tailings are normally discarded as open-circuit tailings and combined with rougher tailings to produce general mill tailings in commercial plant operation. However, the rougher and cleaner-scavenger tailings are almost always kept separate in laboratory testing.

Cleaner Circuit pH and Pulp Density

The most common sulfide gangue in many sulfide flotation circuits is pyrite, and cleaner pulp pH values are usually maintained in a range from 11.0 to 11.5 using lime for pyrite rejection. These pH values may be too high for some ores, resulting in depression of valuable by-products. For these ores, a lower cleaner pH of 10.0 or 10.5 may be appropriate, as well as addition of a pyrite depressant such as sulfites or other polymer depressants. Sensitivity of the cleaner circuit to high pH is determined in a series of cleaner tests that are performed at an optimum regrind size and nominal pH values of 9, 10, 11, and 12. The evaluation of grade–recovery curves from these test series are similar to those described for regrind fineness testing.

The evaluation of cleaner pulp density in laboratory test programs is often difficult because of the limited amounts of concentrate generated and the limited number of flotation test cell sizes available. Cleaner pulp densities ranging from 15% to 20% solids are ideal, but laboratory tests may be conducted as low as 5% solids if low concentrate weight pulls are encountered.

LOCKED-CYCLE TESTING

Locked-cycle tests are performed using rougher and cleaner conditions that have been optimized in batch test work. Each test is a series of batch rougher-cleaner tests (cycles) wherein the middlings products—cleaner tailings and cleaner-scavenger concentrate—from one cycle are recirculated to the next cycle. This type of test is very powerful because it can be used to project continuous metallurgical performance from a series of batch test cycles. The data may be used to project mass and metallurgical balances for plant design or, in special cases, provide a baseline for pilot-plant design and operation.

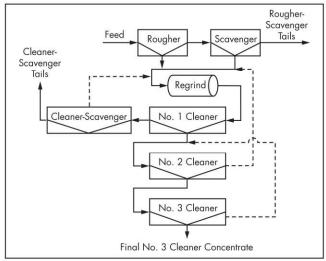
Items of importance to discuss in greater detail are

- Locked-cycle test methodology;
- How to calculate test results, both overall and steady state;
- · The importance of achieving steady state; and
- · How to determine when steady state is achieved.

Figure 11 shows a flow sheet for a typical locked-cycle test, with dashed lines highlighting the recycle middlings products.

Locked-Cycle Test Methodology

A batch test is completed in the first cycle and the middlings streams are stored as slurries. Using Figure 11 as an example, the second cycle would incorporate the cleaner-scavenger concentrate and second cleaner tailings from the first cycle with the rougher-scavenger concentrate from the second cycle for regrinding and cleaning. The third cleaner tailings from the first cycle would be mixed with second cleaner concentrate from the second cycle as feed to the third cleaner. This procedure is repeated for several cycles. The minimum number of cycles required will vary with the ore type and flow-sheet



Adapted from Thompson 2016

Figure 11 Typical flow sheet for a locked-cycle test on a copper ore

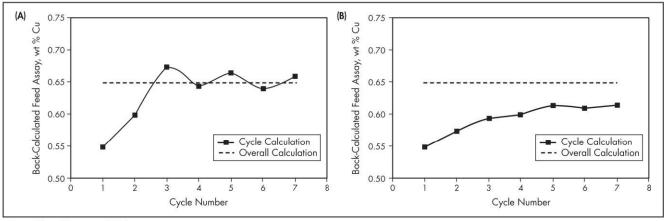


Figure 12 Locked-cycle test (A) achieving steady state and (B) not in steady state

configuration. However, it has been author Thompson's experience that the minimum number of cycles should be equal to the number of cleaner stages plus three. In actual laboratory practice, most tests are performed using seven cycles. Wet filter cake weights of final products from each test cycle provide an indication of when, or if, the test achieves steady state because these weights should stabilize. The final concentrates are usually the most reliable indicator because these are typically the lowest-weight products from the test. The final cycle will include the recycle streams as separate test products in addition to the final products of concentrate and tailings.

The issue of water recycle is rather complicated. It is impossible to simulate plant process water in cycle testing for greenfield projects because these tests do not take into account recycle water aging in places such as thickeners and tailings ponds. If water is an issue (e.g., if seawater, brackish water, or sewage treatment plant effluent is used), then a pilot plant is probably warranted.

Locked-Cycle Calculations

Metallurgical balance calculations for locked-cycle tests are similar to those used in batch tests. Test product weights and assays are used to calculate metal units and distributions for the overall test. The overall metallurgical balance is examined to determine if the final concentrate weights and assays have stabilized in the later cycles, bearing in mind that variations should normally not exceed $\pm 3\%$. If the test has stabilized, then the weights and assays of products from the last three cycles, excluding the recycle products, are used to calculate a "steady-state" metallurgical balance that should simulate continuous operation. The recycle stream weights and assays from the final cycle are then used to calculate the metal distributions in these streams as a percentage of new test feed. They do not enter into the steady-state balance.

Importance and Determination of Steady State

Meaningful data from locked-cycle testing can only be obtained if the test reaches steady state. This implies constancy and is usually achieved in the last three cycles of the test. Inputs equal outputs; for example, if a 2,000-g test feed charge per cycle is used, then the cycle products—concentrate and tailings—should sum to 2,000 g. This concept of input equals output must also hold true for metal balances in

addition to weight (mass) balances. If this is not the case, then there is a buildup of recirculating middlings somewhere in the test flow circuit that must be addressed, usually in subsequent additional test work.

The achievement of steady state may be rigorously calculated by comparing the back-calculated head (cycle feed) assays of the individual test cycles with the overall test backcalculated head, including the recycle middlings from the last cycle. The individual cycle back-calculated heads should intersect the overall back-calculated head several times for the test to be in steady state. Figure 12 shows examples of a test in and out of steady state, respectively.

The achievement of steady-state conditions for ores containing valuable secondary minerals (i.e., molybdenite or gold) is often problematic. The tendency for copper minerals to crowd out the minor minerals can result in the building up of these minerals in middlings streams, particularly in the cleaner-scavenger concentrate.

ORE VARIABILITY AND MODELING

Ore variability and geometallurgy programs are extremely important parts of any flotation process development project. The variability testing is normally started after the rougher and cleaner parameters have been established. Selection of samples for testing may be based on ore types, locations in the ore body, or anticipated mining plan, such as when will a certain section of the ore body become flotation plant feed? These programs integrate mineralogical examination and characterization with comminution testing and flotation testing and distribute the test data across the mineral deposit. A typical ore variability program will involve mineralogical examination, comminution testing, and flotation testing of at least 100 samples, and programs of more than 300–400 samples are not uncommon, depending upon the size of the ore body.

Flotation tests in ore variability programs include batch rougher tests, batch cleaner tests, and locked-cycle tests. Rougher tests include size-by-size analyses of feed, concentrate, and tailings to obtain recovery by size data. A well-planned variability program should include at least 100 rougher tests and 25 cleaner tests. The cleaner tests may be performed on composite samples or selected individual samples and at least two cleaning stages are normally used, though the number of cleaner stages used will depend upon

the results obtained in the batch cleaner optimization study. Locked-cycle tests should be performed on the same samples as the batch cleaners. This will provide a good comparison for upgrading in batch and semicontinuous cleaner tests.

BATCH FLOTATION TESTING FOR FLOTATION CIRCUIT OPTIMIZATION

Traditionally, batch laboratory flotation tests are performed using flotation feed samples and are used as a tool for circuit design or to optimize feed floatability properties. When performed on different streams of a flotation circuit during a circuit survey, they can also play an important role in diagnosing and optimizing a flotation circuit by providing a measure of ore floatability.

The properties that affect the propensity of an ore to float include particle size, mineralogy, degree of liberation, reagent coverage, and the presence of deleterious surface coatings (Trahar and Warren 1976; Sutherland 1989; Crawford and Ralston 1988). Because of system complexity, it is not yet possible to measure ore properties and "calculate" the ore floatability. Instead, it must be inferred from the results of a flotation process. This is not straightforward, as it cannot be determined directly from flotation circuit recoveries because these recoveries are also affected by the operating conditions used in the industrial flotation cells (airflow rate, froth depth, impeller speed, etc.). Batch laboratory flotation testing can provide a benchmarking tool, where operating conditions can be kept constant and differences in flotation response can be used as a measure of ore floatability.

These types of specially designed tests performed in conjunction with a flotation circuit survey can provide measures of ore floatability for use in flotation circuit modeling, as a diagnostic tool to assess flotation mechanisms, or to determine the effectiveness of ore floatability modification strategies (e.g., regrinding, reagent addition). In the following sections, the experimental procedure recommended to perform these tests are outlined and analysis options presented.

Experimental Considerations

Ore floatability characterization batch flotation tests are best performed using samples collected directly from the flotation circuit to be studied. In this way, the characteristics of the particles in the float test feed will truly reflect that in the process. The objective is for the batch flotation feed sample to have the same particle size distribution, mineralogy, degree of liberation, and surface chemistry in the test as that of the circuit stream being studied. Time between sample collection and flotation should be minimized to prevent particle surface oxidation.

The tests should be performed to minimize the impact of operating conditions on the result, using the same air rate, impeller speed, and froth depth in all experiments. Air rates should be high (>10 L/min) to minimize bubble overloading effects that occur where there is insufficient air to remove all particles that are "ready" to float. The impeller speed chosen should be that required to keep all particles in suspension but not so high that it results in particle surface modification by abrasion. The froth depth should be kept to a minimum and the entire froth phase should be removed by scraping at regular intervals during the experiment. Bottom-driven laboratory flotation cells where a fixed depth scraper can remove a constant depth of froth regularly throughout the experiment have proved ideal for doing these types of experiments (Runge 2010). Figure 13 shows examples of modified Agitair-style bottom-driven cells. These types of cells have the advantage that the froth flow is not impeded by the impeller and can be systematically removed using a fixed froth depth scraper to give very consistent results. However, top-driven laboratory machines with scrapers designed to move around the impeller can achieve similar consistencies in performance.

Tests should be performed over a time period sufficient to remove all of the floatable particles in the system. Concentrates should be collected at regular intervals such that a recovery-versus-time curve can be constructed and both the rate of flotation (i.e., how fast the target mineral is recovered)





Figure 13 Bottom-driven laboratory flotation machines



Courtesy of Magotteaux

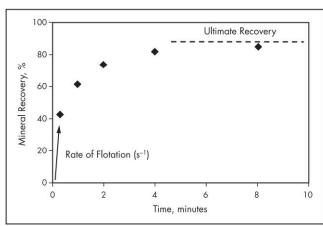


Figure 14 Mineral-recovery-versus-time curve depicting the mineral rate of flotation and the ultimate recovery achievable

and the ultimate recovery achievable can be established with confidence (Figure 14). The test begins when the air is switched on, and the end of the test is dictated by the need for the final concentrate collected to contain no floatable material. In this way, the point at which the recovery time curve asymptotes, which is used to determine the ultimate recovery, can be established with confidence.

As concentrate is removed from the batch flotation cell, it is important to add water to maintain a constant froth depth. This water should have the same chemical composition as that in the original sample to prevent changes in particle surface chemistry. This can be achieved by filtering another sample of the same stream and using the filtrate. Plant process water dosed with an appropriate amount of frother is usually a viable alternative.

Frother concentration needs to be high during the batch test to prevent bubble coalescence and to maintain a constant bubble size in the pulp and a stable froth to minimize particle drop-back. Excessive water recoveries, however, are an indication that the frother concentration is too high. For frothers normally used in flotation, a frother concentration of 30 $\mu L/L$ is usually sufficient to achieve this aim. Frother should be added at the start of the test and also dosed at a similar concentration in the process water added to the cell throughout the experiment. This is because frother concentration tends to decrease with time during a flotation test as it is removed with the concentrate. No reagent, other than frother, should be added to these tests so that the floatability of the particles in the sample is not altered from that of the original stream.

Tests should be performed on all the major streams of the circuit—bank feeds, concentrates, and tailings, and not just the circuit feed. Since viscosity can play a complicating role at high percent solids, streams need to be diluted so that it is only the intrinsic particle floatability that affects the response. Recovery rates can also be lower than they should in concentrate streams performed at high percent solids because there is insufficient air to remove all the particles that would otherwise float. As a rule of thumb, flotation percent solids in the pulp when floating streams collected from the cleaners should be kept below 10% and rougher-scavenger feed and tailing streams kept below 30%.

Analysis Options

The information derived from the previously described types of ore characterization batch flotation tests can be used in a variety of ways, as outlined in the following sections.

Benchmarking

Most plants will be surveyed on a regular basis with a view to either benchmarking performance or testing the effectiveness of a circuit modification. Because of the large number of variables that can impact flotation, it is sometimes difficult to determine the reason for a change in result. Ore characterization batch tests performed on the feed during a survey can establish a measure of ore floatability that can be compared over time. The tests are performed using the same operating conditions so that any differences in a result can be confidently related to changes in grind size, liberation, mineralogy, or surface chemistry. Differences in performance of surveys where the ore floatability is the same can then be attributed with confidence to changes that have occurred within the circuit itself.

Assessment of Particle Modification Processes

Regrinding and reagent addition are employed within a flotation circuit to maximize the recovery and selectivity of the process. Batch flotation tests performed on streams collected before and after these types of processes can be compared to determine their effects on ore floatability (Runge et al. 2004; Runge 2007). This is a fairly simple process when there is only one input and output stream, as in a regrind. Batch flotation tests in this instance can directly be compared. To compare floatability across processes where there are multiple input and/or output streams (e.g., circuit or flotation bank), batch flotation data can be combined by weighting the data according to the relative flow of the mineral in the streams feeding or exiting the process (Equation 1). The aim is to produce a combined feed batch test result to compare to a combined product batch test result.

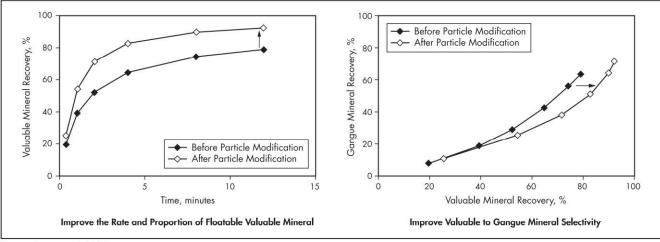
$$R_{\text{mineral }j}^{\text{combined stream, } t \text{ minutes}} = \frac{\sum_{s=1}^{n} F_{\text{mineral }j}^{\text{stream } s} \times R_{\text{mineral }j}^{\text{stream } s, t \text{ minutes}}}{\sum_{s=1}^{n} F_{\text{mineral }j}^{\text{stream } s}}$$
(EQ 1)

where

 $F_{\text{mineral }j}^{\text{stream }s} = \text{flow rate of mineral }j \text{ in stream }s$ $R_{\text{mineral }j}^{\text{stream }s,t \text{ minutes}} = \text{cumulative recovery of mineral }j \text{ in stream }s \text{ after }t \text{ minutes of flotation in the standard laboratory batch flotation test}$

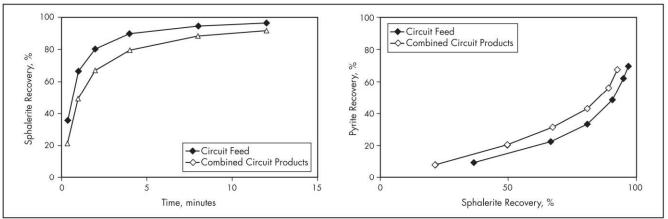
By comparing batch flotation tests before and after a particle modification process such as reagent addition or regrinding, the metallurgist can deduce whether the strategy employed is achieving the desired result. The aim of the modification process is to increase the rate and amount of valuable mineral recoverable while improving selectivity of the valuable mineral with respect to the gangue recovery, as depicted in Figure 15.

These types of comparisons are not only useful to make before and after an individual process or unit, but they can also be used to assess the net effect of all process modification processes within a circuit by comparing the batch test recoveries in the circuit feed and combined circuit product streams.



Source: Runge et al. 2004

Figure 15 Aim of particle modification processes



Adapted from Runge et al. 2004

Figure 16 Comparison of batch flotation test results in the feed and combined product streams of a sphalerite circuit within which lime addition and regrinding were employed, aiming to depress pyrite

In the example shown in Figure 16, batch test comparison was able to show that the lime addition and regrinding that had been employed in a sphalerite flotation circuit to depress pyrite was not successful. Pyrite recovery was depressed by the lime, but the sphalerite flotation was also depressed and to a greater extent than the pyrite, resulting in poorer sphalerite/pyrite selectivity.

The advantage with batch tests is that they enable the ore floatability to be analyzed in isolation to those effects related to circuit operation. What should also be kept in mind is that they are a measurement of pulp floatability only and are not an indicator of full-scale froth-phase performance. In the batch flotation tests, the froth-phase effects have been minimized by using shallow froth depths and by continually removing the froth. Ore properties will affect the froth phase, and this should also be kept in mind when considering the effectiveness of particle modification processes.

Determination of Flotation Mechanisms

Particles can be recovered to the concentrate of a flotation circuit by several mechanisms. They can attach to bubbles because of a hydrophobic surface. They can be locked in composite particles with a hydrophobic mineral and "come along for the ride" (e.g., gangue in unliberated particles, piggy-backing on particle surfaces). Alternatively, they can be entrained unselectively in the water recovered to concentrate.

To maximize the selectivity of the process, it is important to maximize the floatability of the valuable wanted mineral while minimizing the floation recovery of the unwanted gangue minerals. It is important, therefore, to understand the mechanisms of recovery in a floation circuit so that appropriate strategies for improvement can be developed.

Mineralogical analysis and surface chemistry measurements can be employed to judge whether gangue is reporting to concentrate because of composite particles or natural hydrophobicity. It is sometimes difficult, however, to judge the proportion of gangue recovery in a circuit due to entrainment. Batch flotation tests of concentrate streams in a circuit provide a means of making this judgment. Gangue not recovered in these tests is not floatable and can only be present in the concentrate stream because it has been entrained in the flotation bank from which it originates. Figure 17 shows recoveries

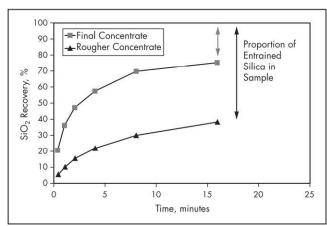


Figure 17 Silica recovery in batch flotation tests performed using a rougher and a final concentrate collected from an industrial copper concentrator

of silica measured in batch flotation tests performed on the rougher concentrate and final concentrate collected from a copper concentrator. It demonstrates how silica recovery in the rougher is largely due to entrainment, whereas in the final concentrate, after re-flotation, the proportion of gangue that does not float is significantly reduced. Excessive entrainment recovery to a final concentrate can be addressed by additional flotation stages or dilution of pulp density.

Batch flotation test data can also be used to assess valuable mineral recovery (or non-recovery) mechanisms. Fast flotation kinetics of the valuable mineral in a tailing stream is usually indicative of insufficient flotation capacity or poor flotation cell operation (e.g., use of very deep froth depths in scavengers). Slow kinetics for the floatable mineral, exhibiting poor selectivity with gangue, will be observed when the grind size is too fine (something that can occur after regrinding in a cleaning circuit). Low valuable mineral recovery in the tailing is an indication that improvements in circuit recovery will best be achieved by addressing ore floatability—either by improving flotation selectivity, improving the feed liberation, or addressing flotation chemistry issues.

Flotation Model Development

Flotation recovery and separability will be a strong function of the circuit arrangement, flotation bank residence times, ore floatability, and the cell operating parameters. Flotation modeling provides a tool for optimizing these various interactive effects and enables simulation of different strategies for circuit improvement.

Harris et al (2002) has summarized a methodology that uses ore floatability characterization batch tests performed on different streams of a floatation circuit as a way of deriving parameters of a floatability component model for use in circuit simulation. Many case studies have been published in the literature, highlighting the use of this methodology for floation circuit optimization (Crosbie et al. 2009; Schwarz and Alexander 2007).

In floatability component models, the ore floatability of different minerals in different size classes is represented by fast, slow, and non-floating components. Because the batch flotation tests are all performed using the same operating conditions, any change in response in the different streams is due to a change in ore floatability. A model is fitted to the batch test data to determine the proportions and rates of the fast, slow, and non-floating components in the feed and all of the streams of the circuit. The data can also be used to develop models to modify ore floatability at points in the circuit where regrinding or reagent addition occurs.

Performing batch flotation tests during a circuit survey not only provides useful data for circuit diagnosis but also means that the information required to develop models of the circuit using the above techniques is collected. Without this type of ore characterization data, it is very difficult to isolate cell operating effects (e.g., poor froth recovery, insufficient air) from ore floatability effects (i.e., selectivity and recovery potential) from circuit survey data alone.

SUMMARY

The key concepts underlying a laboratory test program for the development of a copper sulfide flotation process are described. The testing program includes scoping flotation tests for reagent scheme selection, rougher flotation parameter evaluation, cleaner circuit evaluation, locked-cycle testing, and ore variability and modeling. The concepts explored can be applied or modified for most sulfide flotation systems.

Ore characterization batch tests for flotation plant optimization are relatively simple to perform and produce very repeatable results if performed using the systematic procedures outlined in this chapter. If performed routinely during circuit survey work, they provide important information about how the variables that affect ore floatability (size, mineralogy, chemistry) are impacting flotation circuit performance. They provide benchmarking of ore floatability between surveys and a diagnostic tool for offering information about recovery mechanisms or the effectiveness of particle modification processes. They can also be used to develop floatability component models that can simulate options for improving the grades and recoveries achievable in the circuit.

It is recommended that ore characterization batch flotation tests are performed in conjunction with other measurements. Although batch laboratory flotation tests indicate the overall result of a change, other information from sizing, mineralogical, and chemical analysis tools can be used to determine the reason for a particular result.

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