CHAPTER 7.8

Non-Sulfide Flotation Testing

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With the exception of a few elements, such as the base and precious metals, which are predominantly obtained from sulfide minerals in sulfide ores, most other elements or their minerals are obtained from non-sulfide ores. Thus, non-sulfide mineral flotation from ores covers a wide variety of minerals of industrial importance. The term *non-sulfide minerals* (or *ores*) comprises minerals (or ores) belonging to the following classes: oxides, silicates, sulfates, phosphates, carbonates, and halides. These are variously described in the industry under terms such as *industrial minerals*, *nonmetallic minerals*, *construction materials*, *functional minerals*, and so on. Examples of important non-sulfide mineral ores include phosphate, iron oxides, kaolinite and bentonite, spodumene, potash, borates, trona, fluorite, calcite, dolomite, limestone, barite, mica, feldspar, quartz, silica sands, monazite, kyanite, magnesite, chromite, bauxite, ilmenite, rutile, manganese oxides, graphite, talc, and cassiterite. Strategies and approaches for laboratory and pilot-plant testing are discussed in this chapter using examples from flotation beneficiation of well-known commodities such as phosphate, iron ore, clays, potash, and feldspar. Details of the processing flow sheets are discussed elsewhere in separate chapters devoted to each mineral commodity. Historical aspects of the development of non-sulfide flotation can be found in Fuerstenau (2007), Nagaraj and Ravishankar (2007), and Nagaraj and Farinato (2016).

Flotation concentration of non-sulfide minerals is significantly more complicated than that of sulfide minerals. This complication arises primarily because of the similarities in surface chemical composition and properties in an aqueous medium between non-sulfide value minerals and non-sulfide gangue minerals. In sulfide mineral flotation, the primary separation task comprises selective flotation of sulfide minerals from the vastly different non-sulfide minerals. In non-sulfide flotation, the task is floating one non-sulfide mineral from many other non-sulfide minerals. This separation task requires significant know-how and art to manipulate and exploit small differences in surface properties between minerals and to develop specific process conditions. Redeker and Bentzen (1986) noted that “flotation of nonmetallic minerals is sometimes viewed as alchemy. However, the art is, in reality, based on sound chemistry, experience and a little luck in the treatment of new ores. In the flotation of nonmetallic ores, as opposed to base metal sulfides, highly specific treatment conditions are required to accomplish separations.” A vast amount of published literature exists for both the fundamental and applied aspects of non-sulfide flotation, beginning with the pioneering work of U.S. Bureau of Mines investigators in the late 1920s (Coghill and Clemmer 1935); however, this literature is far more fragmented than that for sulfide flotation (Aplan 1994). This is necessarily so if one considers the variety of non-sulfides that are beneficiated in the industry and the artisanship that surrounds these processes. Separation schemes for non-sulfide minerals are distinctly different from those for base metal sulfide minerals. Such distinction can be readily understood by the fundamental differences that exist in physical and chemical properties between sulfide and non-sulfide minerals (see Nagaraj and Ravishankar 2007).

Plant practice is often consistent with the major differences between sulfides and non-sulfide minerals. Aplan and Fuerstenau (1962) noted that “selective flotation in non-sulfide systems is often difficult in that one class of compounds alone, the carboxylic acids, may float nearly the entire array of non-sulfide minerals under the appropriate conditions. The non-sulfide mineral flotation operator is confronted with a vast array of modifying reagents.” This is in contrast to sulfide mineral flotation where a wide variety of collectors, many of which exhibit a high degree of selectivity for a given sulfide, is available (and used) for the relatively small number of sulfide minerals floated. Modifiers are thus far more critical to successful separations in non-sulfide systems than they are in sulfide systems.

Given the diversity of non-sulfide flotation plant practice and the rather fragmented literature information, a practitioner new to non-sulfide flotation may initially find the task of developing a laboratory program and flow sheet rather bewildering and daunting. However, this is certainly not the case. A

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common theme and a set of guidelines emerge for non-sulfide flotation if one considers the types of reagents used (both collectors and modifiers) and the types of treatment methods and conditions employed. This chapter aims to provide basic guidelines for laboratory and pilot-plant flotation testing for a given non-sulfide ore flotation system to assist in the following goals:

1. Develop a new flow sheet or evaluate an existing one.
2. Optimize and improve performance of an existing plant process via a better reagent and/or engineering schemes.
3. Evaluate different ore types and ore variability.
4. Develop process models.
5. Produce sufficient samples for market utilization studies.

These guidelines are based on information gathered from published literature on both fundamental and applied aspects; articles describing plant practices in trade journals, conference proceedings, and operators' forums; and experiential knowledge of metallurgists. A compilation of features of the major industrial non-sulfide mineral flotation separations is provided in Table 18 of Chapter 7.5, "Flotation Chemicals and Chemistry," and in the following selected references: Aplan and Fuerstenau (1962); Arbiter and Williams (1980); Aplan (1994); Baarson et al. (1962); Coghill and Clemmer (1935); Cytec (2010); Davis (1985); Falconer and Crawford (1947); Falconer (1961); Fuerstenau (1962); Fuerstenau et al. (2007); Gaudin and Glover (1928); Holme (1986); Houot (1983); Jones and Oblatt (1984); Malhotra and Riggs (1986); Manser (1975); Miller et al. (2002, 2007); Mulukutla (1994); Peres et al. (2007); Redeker (1981); Redeker and Bentzen (1986); Somasundaran and Moudgil (1987). A few useful references for laboratory testing include Dunne et al. (2013); Macdonald and Brison (1962); Spedden (1985); and Williams et al. (2002). North Carolina State University Minerals Research Laboratory reports are also an excellent source for laboratory testing and development for a variety of non-sulfide mineral flotation systems (NC State University 2018).

The guidelines serve to establish a starting point and a basis for designing the laboratory program; the metallurgist still requires empirical testing to refine the reagent schemes, conditions, and flow sheets in an iterative manner to meet metallurgical and economic objectives. The pilot test methods presented in this chapter can be used to establish suitable flotation circuits. Analytical techniques employed to gather meaningful information from test work data are discussed in this chapter as well.

DISTINGUISHING FEATURES OF NON-SULFIDE FLOTATION

Several important distinguishing features of non-sulfide mineral systems, in comparison to sulfide flotation systems, dictate strategies and approach to laboratory flotation. These are listed in Table 1.

Table 1 Non-sulfide versus sulfide mineral flotation distinguishing factors*

Factor	Non-Sulfide Mineral System	Sulfide Mineral System
Ore type and mineralogy	Very diverse and broad classes (e.g., silicates). Value minerals and gangue minerals are both non-sulfides and have rather similar surface properties; thus, separation selectivity is a challenge (ores may contain gangue sulfide minerals such as pyrite, but these are easily removed using traditional sulfide collectors prior to non-sulfide mineral separation). Careful selection of collectors, modifiers, and pulp conditions is necessary. Many ores are the sedimentary type and contain a significant amount of slimes requiring desliming. Minerals can have a wide range of substitutions, compositions, and structures (e.g., the silicate minerals)—all of which can influence surface properties.	Value minerals are invariably sulfides and precious metal minerals. The majority of gangue minerals are non-sulfides; only a few gangue sulfide minerals are present. Collectors are invariably selective and specific for sulfide minerals; selection of modifiers and conditions are relatively more straightforward. Ores are predominantly igneous type.
Surface chemistry in the aqueous phase	Relatively small differences between a value mineral and the gangue. Surface chemistry is dominated by reactions involving H^+, hydroxyl (OH^-), and the lattice ions. All of the minerals in the system undergo strong surface charging, hydration, dissolution, and hydrolysis—all of which influence surface chemistry. Various species released from non-sulfide minerals can have complex interactions in the aqueous phase; the products of these processes, in turn, can interact with many minerals causing changes in flotation selectivity. Redox reactions, and complications arising from these, are absent in most systems (most minerals are insulators).	Very large differences between value and gangue minerals. Surface chemistry is dominated by complex multistep redox reactions (most sulfides are excellent conductors); these can have a significant adverse effect on flotation. Relatively smaller contribution from hydration, hydrolysis, and dissolution.
Water quality	Critical to achieving selectivity of separation. Imperative to conduct final testing in water from a source representative of local water to be used. Any change of water source during operations should be checked thoroughly before general use to determine its effect on selectivity and reagent consumption.	Not as critical; effects are a matter of degree rather than catastrophic.
Comminution	Grinding for liberation is not always necessary. In some systems, fine grinding is necessary to achieve adequate liberation; this results in substantial amounts of slimes. Many ores are from sedimentary deposits and can be processed without any comminution steps (e.g., phosphate, glass sands, clays, and potash ores). Liberation of value minerals from gangue is not a major limiting factor in these systems.	Grinding is critical to achieving liberation. Liberation is most often incomplete and/or inadequate.
Flotation conditions and strategy	Highly specific treatment conditions are frequently required to achieve adequate selectivity and satisfactory metallurgy, the most critical aspect of which is generally the quality and purity of the finished mineral product (which can go directly to use in many cases).	Treatment conditions are common to almost all of the systems. The mineral product is used for metal production.

(continues)

Table 1 Non-sulfide versus sulfide mineral flotation distinguishing factors* (continued)

Factor	Non-Sulfide Mineral System	Sulfide Mineral System
Flotation conditions and strategy (continued)	Both direct flotation (flotation of targeted value mineral away from gangue) and reverse flotation (flotation of gangue minerals away from value mineral) are plausible and frequently used options. The choice is dictated by the efficiency and selectivity of separation, composition, and mineralogy of the ore, choice of reagents, pulp conditions, and economics.	Direct flotation of sulfides is the most common practice.
	Flotation rates and particle sizes floated are large in some systems (e.g., phosphate ore, potash, and silica); floated particles must be collected quickly to prevent drop back. Some plants even refer to their process as *flash flotation*, likening it to a flash distillation process. For example, in phosphate flotation, retention times are a few minutes. (Of course, there are systems wherein the flotation rates and particles sizes are quite small, requiring long residence times [e.g., flotation of fine anatase from fine kaolin clays; residence time could be 30–60 minutes]; these systems are similar to the sulfide ore systems.)	Particle sizes are typically <212 μm. Slow flotation rates and long residence times are common.
Pulp preparation	Pulp preparation prior to flotation separation is critical in non-sulfide separations because of complex surface interactions. This includes processes such as washing (acid or caustic), scrubbing or attrition scrubbing, desliming, hot pulp conditioning, and high solids conditioning.	No special pulp preparation is required. High solids and high-temperature conditioning are rare. Most operations are at ambient temperature.
	Conditioning: A wide spectrum of conditioning requirements exists for reagents with respect to time, pulp density, agitation intensity, pH, order of reagent addition, types of modifiers, temperature, and water quality. Preconditioning requirements are often less stringent in amine flotation, in contrast to fatty acid flotation. Conditioning in amine flotation is usually at flotation pulp density, unlike in fatty acid flotation.	Short conditioning times are typical (from a few seconds to a few minutes). High intensity and high solids conditioning are rare.
	Desliming: Very common and often a critical step. Some non-sulfide separations are virtually impossible if the feed to flotation is not properly deslimed. The slimes[†] (clays, iron oxides, and hydroxyoxides, etc.) can consume collector, coat coarse particles, cause stable froths with reduced drainage, increase pulp viscosity, and seriously interfere in separations in other ways. The presence of slimes can reduce the ability of collectors to differentiate among various mineral surfaces. Because the slime fraction is usually discarded, it can account for a considerable loss in value minerals. The deslimed material is sometimes separated into coarse and fine fractions and treated separately to optimize flotation conditions for each size fraction.	Desliming is not a common practice (used only in a few niche systems).
Circuit complexity	Flotation circuits vary widely and can be rather complex, some involving several stages of sequential flotation separation, each stage having its own specific treatment conditions and preparation requirements. Plant flow sheets often employ other separation techniques prior to, within, and after flotation circuit to reach the target grade (e.g., gravity, magnetic, electrostatic, selective flocculation, air classification, and further comminution and sizing); specific examples are found in other chapters. Strategies employed in flotation may be influenced by separation units prior to and after flotation. High circulating loads and water reuse may not be possible in many non-sulfide ore systems, for example, the three stages of flotation in feldspar beneficiation, each stage requiring dewatering, washing, and use of fresh/clean water for the next stage.	Flotation circuits tend to be similar in most of the systems and relatively less complex. High circulating loads and water reuse are typical in all circuits. Occasionally, magnetic separation may be used. Gravity separation is used for ores containing gold and for platinum group metals.
Feed characteristics	Unlike sulfide ores, the concentration of valuable minerals in non-sulfide ores is typically high (e.g., kaolin clays, phosphate, iron ore, glass sands, feldspar, barite, potash, and others); however, tonnages processed are generally lower than those in sulfide ore systems.	Low concentration of valuable minerals in most ores. Very large tonnages (e.g., 100,000–300,000 t/d in copper ore operations).
Reagents	The large concentrations of valuable minerals, and their significant hydrophilicity (high degree of hydration and hydrogen bonding), typically require high dosages of long-chain collectors; for example, 500–1,000 g/t of C_{18} fatty acid in some systems (C_{12} considered minimum chain length). In some systems, collector adsorption is via metal complex formation and surface precipitation; in others, it is electrostatics. Mineral lattice anion has a greater role than metal ion (in contrast to sulfides). Modifiers are critical to achieving required selectivity and efficiency. Some separations are impossible without appropriate modifiers. Multiple modifiers may be required in some systems. Their dosages also tend to be high.	Very low dosages (2–50 g/t) and short-chain (C_2–C_5) collectors. Sulfide surfaces are generally far less hydrophilic (weaker hydration and hydrogen bonding). Role of modifiers is not as critical as in non-sulfide systems. pH modifiers are most common. Other modifiers are used as needed. Collector adsorption is dominated by coordination covalent bonding; metal ion of mineral is very important.
	Reagent solubility is often an issue requiring specialized preparation procedures prior to use. Examples of this include neutralization of amine collectors, emulsification of fatty acids, and high-temperature solubilizing of organic polymeric depressants.	No special equipment is necessary for reagent dosing.

*More specific information and details can be found in Aplan and Fuerstenau (1962); Aplan (1994); Baarson et al. (1962); Coghill and Clemmer (1935); Cytec (2010); Davis (1985); Falconer and Crawford (1947); Falconer (1961); Holme (1986); Malhotra and Riggs (1986); Manser (1975); Miller et al. (2002, 2007); Mulukutla (1994); Peres et al. (2007); Redeker (1981); Redeker and Bentzen (1986); Somasundaran and Moudgil (1987). Factors such as froth stability, pulp density, and others are not highlighted and distinguished because the ranges and observations appear to be common to both sulfide and non-sulfide flotation.

[†] The term *slimes* has a loose meaning in non-sulfide separation and is system dependent; for example, −100 μm particles are considered slimes in some phosphate operations, whereas in other systems, particles <1–10 μm are considered slimes.

STRATEGY AND APPROACH TO LABORATORY TESTING

The strategy and approach to laboratory testing are dictated by whether the goal is to improve efficiency and selectivity in an existing operating plant or to design a flow sheet for a new ore or project. The key aspects are given in Table 2.

Preparation Prior to Laboratory Testing

The following items need to be addressed prior to laboratory testing to ensure that the outcomes are meaningful:

- Appropriate selection of samples for project development
- Proper collection of samples for plant optimization testing
- Proper subsampling and preparation of samples for flotation testing
- Understanding of the mineralogy and liberation characteristics of the sample collected for testing
- Appropriated analytical procedures and incorporation of a suitable quality assurance and quality control program
- A robust design for laboratory tests that will provide statistically meaningful outcomes

Laboratory Test Work

If grinding is involved, a grinding study is performed to develop the appropriate procedure for grinding the feed to desired liberation (determined by mineralogy and economics) while maintaining as narrow a size distribution as possible. This is typically done using a laboratory rod or ball mill (ceramic grinding media used in systems where iron species create a problem). Size-by-size mineralogical analysis is then conducted to determine the mineral separation potential, valuable mineral content, the type of gangue minerals in the slime (or fine size) fractions, and the need for desliming to reject predominantly problematic gangue minerals with an acceptable loss in values. Desliming can be performed using hydroseparators (elutriators, desliming thickeners), cyclones, sieves, or simple sedimentation equipment (e.g., differential settling in a tall cylinder or a tank with a conical bottom). If aging of the pulp is an issue, then it is best to prepare the flotation feed charge prior to the flotation test.

Water Quality and Water Chemistry

Water quality and water chemistry in non-sulfide flotation are especially important because aqueous species—both anionic species and multivalent metal ions—interact with each other, with non-sulfide mineral surfaces, and with reagents via both chemical (e.g., calcium on phosphate mineral or carbonate mineral dictated by solubility products) and electrostatic pathways (calcium on quartz) to influence reagent adsorption and selectivity of separation. These aqueous species may be from deliberately added modifiers, from the water source available at the mine site, or from various minerals (both value minerals and gangue) in the system. Multivalent ions (both anionic and cationic) can be either beneficial or detrimental depending on the specific system in question and the type of collector considered. Species present in the water source and/or released from minerals (because of their finite and significant solubility) are called *unintended species* or *modifiers*. These can have significant adverse effects on selectivity and recovery, especially when water is recycled and circulating loads are present.

Although the effects of species from deliberately added (or intended) modifiers are reasonably well-known and targeted for a specific function, those of unintended species generated from minerals in the ore are less well-known and predictable. Unintended species can have a wide range of effects on both efficiency and selectivity of separation:

- They can interfere with collector adsorption on desirable minerals (e.g., Ca^{2+} adsorption on quartz can prevent amine collector adsorption; sulfate, SO_4^{2-}, ions in water can adsorb on barite and prevent anionic collector adsorption).
- Flotation of targeted minerals can be reduced.
- They can promote collector adsorption on, and flotation of, undesirable mineral(s) via inadvertent activation (inadvertent Ca^{2+} adsorption on gangue quartz to promote fatty acid adsorption and flotation). The pH of activation by metal ions is strongly influenced by the type of metal ion and the pH of formation of first hydroxide (or hydroxyl) species.
- Froth characteristics can change (highly variable and difficult to predict; froth can be influenced by the presence of organics and/or gelatinous/colloidal precipitates in the aqueous phase).
- Pulp viscosity can change (multivalent ions can stabilize/promote mineral network or form colloidal or gelatinous precipitates).

Inadvertent activation, for example, is often difficult to predict, because it is strongly influenced by mineralogy and pulp conditions that exhibit significant variability in a plant. Indeed, one of the main functions of modifiers deliberately added (e.g., lime, soda ash, sodium silicate, polyacrylates, sodium sulfide) is to control the effects of inadvertent and unavoidable species. Locked-cycle testing should be considered if water chemistry effects are anticipated (e.g., when the quality of the available water source is less than desirable, or when flotation conditions such as low pH cause excessive release of species from some minerals). A good example of the influence of water chemistry is the selective flocculation desliming of quartz from hematite. The presence of calcium and magnesium ions in iron ore pulps that are upgraded by selective desliming can result in indiscriminate flocculation of both iron oxide and siliceous gangue slimes and/or in interfering with amine adsorption on quartz. In other cases where fatty acids are used as collectors, cations such as Ca^{2+}, Mg^{2+}, and others (Fe^{3+}, Al^{3+}) will react with fatty acids to form insoluble compounds thereby consuming the collector (in some cases, metal soaps can precipitate on or near the mineral surface, and this can enhance hydrophobicity). In the case of phosphate ores containing apatite and calcite/dolomite, the surface of apatite can be converted to calcite and vice versa depending on pH and carbonate concentration. The dissolved mineral species can alter the mineral surface properties to such an extent that surface properties of various minerals become indistinguishable, leading to loss of selectivity in the adsorption of reagents.

In systems involving sequential flotation of gangue minerals with specific treatment conditions and collector types, the value product from each stage may require dewatering, thorough washing with fresh water, chemical treatment (sulfuric acid, bleach, etc.) to generate fresh surfaces, and use of fresh water in conditioning prior to addition of the collectors (e.g., beneficiation of beryl, spodumene, feldspar, phosphate).

Table 2 Flotation aspects for defining a non-sulfide mineral flotation strategy

Flotation Aspect	Description
Mineralogy	Mineralogical characterization is one of the most important aspects of determining the most effective approach to laboratory flotation of non-sulfide minerals. This includes mineral phase identification, mineral association, and liberation characteristics (if comminution is necessary). This can be accomplished by a combination of popular techniques, such as MLA (mineral liberation analyzer), QEMSCAN (Quantitative Evaluation of Minerals by Scanning Electron Microscopy), XRD (X-ray diffraction), SEM-EDX (scanning electron microscopy–energy dispersive X-ray), EPMA (electron probe microanalysis), optical microscopy, and elemental analysis. Information on impurity or gangue minerals, even if present in minor amounts (e.g., phosphate mineral in iron ores; Fe-Ti minerals in kaolin clays), will be necessary because their presence can have a large impact on the selectivity of separation and final product specification.
Literature review	A survey of plant practice or even past laboratory investigation on ores similar to the one under consideration provides a general framework. Very few ore types have not been explored by mineral separation technologists; strategies for successful separation can generally be gleaned from other operations with similar ores. Many sources for such information can be found, including Chapter 7.5, "Flotation Chemicals and Chemistry," and other chapters on individual mineral commodities in Part 12 of this handbook. These should serve as a starting point for working with a new ore at a bench scale.
Comminution	The need for, and extent of, comminution is dictated by rock or ore type, mineralogy and liberation characteristics, economics, and the grade specifications for the final mineral product. Some ores, such as sedimentary phosphate ores of central Florida (United States), do not require grinding for liberation, whereas hard taconite in northern Michigan (United States) can require extensive grinding with a liberation size of <25 µm (Kawatra and Carlson 2013; Siirak and Hancock 1988). Some ores may require the breaking of aggregates in the mined ore, which could be accomplished in high-shear, high solids conditioning; for example, kaolin clays of Georgia (United States).
Floated mineral(s)	The decision to consider either direct flotation or reverse flotation is largely dictated by the following: • The ratio of the amount of value mineral to gangue mineral. A small ratio favors direct flotation; a very large ratio favors reverse flotation; and for ratios in the intermediate region, either direct flotation or reverse flotation is an option. The decision will be based on economics, selectivity, overall recovery, and the level of complexity involved. • Gangue mineralogy. A sequential reverse flotation scheme to remove one or more impurities in each stage (e.g., feldspar flotation) can be considered when the gangue composition is complex, the ratio of value to gangue is large, and when the different gangue minerals cannot be floated selectively and effectively with the same reagent scheme. In some cases, even if the ratio of value to gangue is large and there are several gangue minerals to be depressed, direct flotation may be considered because a robust collector-modifier scheme is available for selective separation (e.g., direct flotation of diaspore from bauxite ores). Often, ores have gangue sulfide impurities such as pyrite; these can be easily removed by flotation with traditional sulfide collectors before non-sulfide mineral separation. Sometimes sulfides can also be removed by gravity separation. Any gangue talc can be removed easily by flotation with only a frother. • Economics and complexity. The decision to select direct or reverse flotation is dictated by overall economics and complexity of the flow sheet, number of unit operations and separation schemes (magnetic, flotation, gravity, etc., involving isolated water streams) required for each route. If a practical, economical and robust reagent scheme already exists for one route, then this may be preferred to developing a new scheme, which may be expensive and time-consuming unless the new scheme is potentially less complex and more economical. It is often preferable to remove gangue and impurity minerals by physical separations such as magnetic separation, size, and gravity separation prior to flotation if at all possible. This strategy reduces the complexity in flotation. • Product specifications (for either internal use or external sale). This is extremely important because many consumers will require a specific grade or absence of specific impurities. If this target cannot be reached, there is no need to further pursue the project.
Reagent treatment schemes	Reagent and treatment schemes are discussed in Chapter 7.5, "Flotation Chemicals and Chemistry," and in other chapters on individual mineral commodities in Part 12 of this handbook. The choice is dictated by whether direct or reverse flotation is used. Temperature is an important factor to consider when evaluating reagent schemes (e.g., fluorite, kaolin clay, and rare earth ores). Well-established and reputed reagent suppliers have expertise beyond the solutions suggested in the literature. Efforts must be made to simplify the reagent schemes as much as possible (removing gangue by other physical separation techniques can help).
Water availability	Water quality and chemistry have a critical influence (far more than in sulfide ore systems) on both selectivity of separation and recovery of value minerals. Knowledge of the type and composition of the water resources available at the mine site is necessary before any laboratory investigation. If only seawater is available, the laboratory strategy should be developed using seawater as a water source. If fresh water is available, extensive locked-cycle and piloting are necessary to ensure that the water circuit can be closed without interfering with plant performance. Water softening agents may be used for separations sensitive to water chemistry.

In these complex circuits, efforts must be made to isolate water from each circuit and prevent cross-contamination.

BENCH-SCALE FLOTATION EQUIPMENT

This section discusses the equipment necessary for a bench-scale flotation program.

Flotation Cells

The laboratory procedures and equipment designed for conventional flotation have remained substantially the same over the past 50 years, consisting primarily of subaeration-type flotation machines with induced air delivery such as the Metso Denver D12 and the Wemco Mineral-Master 600, or with forced air delivery such as the Essa FTM100 and the Agitair LA-500. Some improvements have been made to laboratory equipment design; however, these have mainly been for ease of use.

Conditioning

Pulp conditioning can be done either in the flotation cell (common when grinding is involved and/or attrition scrubbing is not necessary; e.g., for igneous ores) or in a separate vessel such as an attrition cell or attrition scrubber or attrition-conditioning cell (e.g., for sedimentary ores, no significant grinding is involved). Grinding achieves (1) size reduction, (2) liberation, and (3) generation of fresh surfaces. Attrition-conditioning provides the necessary high-shear scrubbing intensity, with minimal size reduction, to remove surface coatings and expose fresh mineral surfaces for the reagents. Attrition cells can also break up aggregates and disperse minerals effectively. They typically operate at high solids loading with sufficient water to fluidize the solids, typically 50%–80% depending on the system and the pulp viscosity. Small amounts of slimes generated in this operation are often discarded prior to reagent

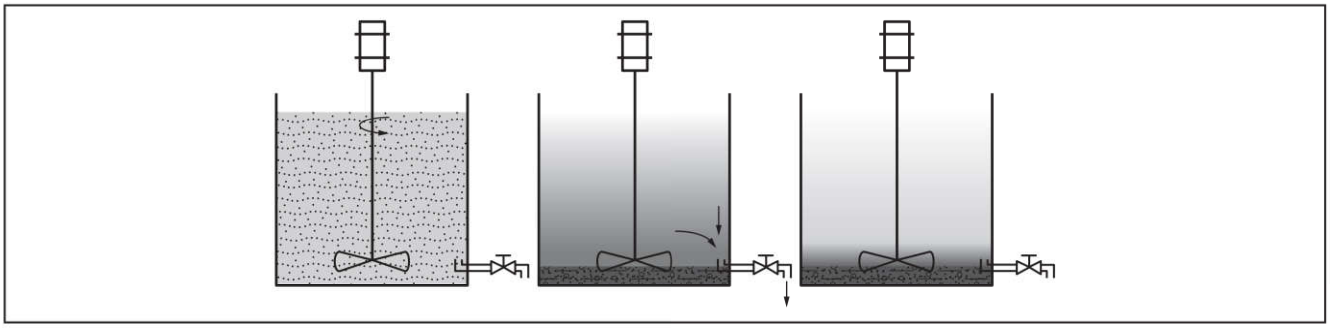

Figure 1 Laboratory desliming by differential settling

conditioning. This includes conditioning with a modifier and a collector. Modifiers could include a dispersant, a pH modifier, an activator or a depressant. Attrition cells can be used in both chemisorbing collector systems (such as fatty acids and hydroxamates) and physisorbing collector systems (such as amines and petroleum sulfonates). Some flotation machines are designed to be used as a separate conditioning device by changing the rotor, removing the stator(s), and using a cell of different geometry (e.g., cylindrical, hexagonal, or square cross-section). Separate laboratory conditioning machines can also be made from a standard drill press, a shaft impeller, and a cylindrical vessel (2 L is typical) mechanically held to the drill press table.

Desliming

Desliming by wet screening is a very common technique because it is easy and does not require special rigs. However, it is slow if material below ~38 µm (especially below 20 µm) has to be removed. The coarser fractions are first removed to protect fine screens and to reduce the amount of screening time. Consistency is hard to obtain with this method because of the varying techniques, water flow rates, and screening times that an operator might use. Differential settling is a straightforward and repeatable way to deslime a feed. It involves agitating a slurry containing slimes at approximately 5%–10% solids, allowing the coarse solids to settle for a set period, then siphoning or using a valve to drain the supernatant water containing slimes from the vessel (Figure 1). This can be repeated several times depending on the degree of desliming required. In-cell elutriation is often the quickest and easiest way of desliming a feed. It is performed by agitating the feed charge containing slimes in a float cell or attrition cell, then injecting water at a constant and consistent rate at the bottom of the float cell. Coarse particles settle and remain at the bottom, and the slimes leave the cell through the overflow launder. Dispersants, such as sodium silicate, sodium polyphosphate, or low-molecular-weight synthetic polymers, can be added to increase the effectiveness of all of these techniques.

Laboratory cycloning is an easy way of replicating many industrial desliming processes. An example of a bench-scale cyclone desliming unit is shown in Figure 2. The unit consists of a feed tank and a cyclone matched by flow characteristics to a feed pump. The slurry is fed into the feed tank, and the pump speed is adjusted to provide the optimum size cut. Once the optimum pump speed is established, the cyclone overflow is collected while water is added to the feed tank to maintain its level.

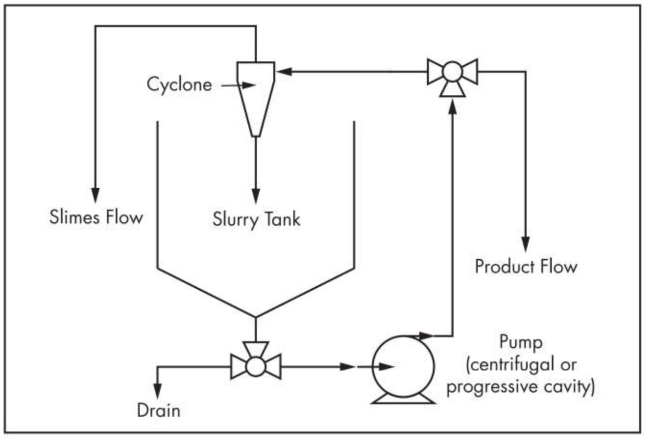

Figure 2 Bench-scale cyclone desliming unit

Bench-Scale Flotation Guidelines

Every ore presents its own challenges and requires a different series of steps and reagents to maximize metallurgical and economic performance. However, most bench-scale flotation experiments for non-sulfide minerals involve both conditioning and flotation. Basic considerations for laboratory testing are given in Table 3.

Flotation is typically carried out to completion. Most laboratory rougher flotation tests take less than 10 minutes for non-sulfide minerals. Scavenger and cleaner tests take approximately 60% of the time that a rougher flotation test takes. Table 4 shows optimum flotation times for laboratory rougher flotation of non-sulfide minerals with a comparison to plant scale.

LABORATORY FLOTATION TEST PROGRAMS

Design of Laboratory Tests

In designing a flotation test program, characteristics of the ore, empirical knowledge of related flotation separations, mineral product specifications, and economic considerations form the basis in the selection of reagent schemes and specific treatment conditions. As in any mineral processing operation, laboratory testing is also essential in overall risk assessment and in identifying potential problems that may be encountered when scaled up to the plant scale. This is well captured in Arthur Taggart's quote: "Make your mistakes on a small scale and make your profits on the large scale."

Rougher and cleaner or rougher and scavenger flotation tests are conducted to evaluate the major variables: grinding

Table 3 Cell size and flotation parameter considerations

Cell Volume, L	When to Use	Flotation Parameters	Examples of Use
0.5–1.5	• Finely liberated ores (<200 µm) • Limited availability of feed material (water and ore) • Valuable mineral constitutes more than 30% of the feed • Slurry solids 20%–25% typical but can range from 10% to 50% for certain applications (see literature for more details) • Scavenger and cleaner evaluation	• 200–500 g feed charge • 900–1,200 rpm • 3–5 L/min airflow	• Phosphate reverse flotation for carbonaceous ores • Finely liberated hematite • Scavengers and cleaners for most non-sulfide ores
2.5	• Most commonly used cell size • Feeds with top sizes <400 µm • Valuable mineral constitutes more than 10% of the feed • Slurry solids 20%–25% typical but can range from 10% to 50% for certain applications (see literature for more details) • Scavenger and cleaner evaluation	• 500–1,500 g feed charge • 1,000–1,400 rpm • 4–7 L/min airflow	• Rougher flotation of iron ores containing >30% Fe • Rougher flotation of phosphate ores containing carbonates and clays • Scavenger and cleaner flotation for coarse phosphate ores
3+	• When sufficient feed is available • Feeds with top sizes >400 µm • Valuable mineral constitutes <10% of the feed • Slurry solids 20%–25% typical but can range from 10% to 50% for certain applications (see literature for more details)	• 1–2 kg feed charge • 1,200–1,900 rpm • 5–10 L/min airflow	• Coarse sedimentary phosphate ores • On-site testing of any ore where ample supply of materials is available

Table 4 Laboratory rougher flotation times and plant-scale comparison

Mineral	Laboratory Flotation Time, minutes	Industrial Flotation Time, minutes
Barite	4–5	8–10
Calcite (from phosphate ore)	1–3	3–6
Coal	2–3	3–5
Feldspar	3–4	8–10
Fluorspar	4–5	8–10
Phosphate	1–3	4–6
Potash	2–3	4–6
Sand	3–4	7–9
Silica (from iron ore)	3–5	8–10
Silica (from phosphate ore)	2–3	4–6

Adapted from Kawatra 2011

stage factors such as fineness of grind, pulp density, type of grinding medium, and chemical additives; conditioning factors such as collectors, depressants, pH, time, intensity, and temperature; and flotation factors such as type of cell, collectors, frothers, depressants, pH, intensity of agitation, and time. Other variables may include desliming, magnetic separation, washing, and in continuous or locked-cycle tests, circulating middlings and water in grinding and flotation.

A laboratory test program can range from a simple flotation optimization for an existing operation to a full greenfield process development. Laboratory flotation testing should be considered in two stages: diagnostic and optimization.

Diagnostic Testing Stage

Diagnostic testing has three important objectives:

1. Determine the viability of flotation separation for the particular ore.
2. Determine the suitability of the ore sample for laboratory flotation testing.
3. Establish a benchmark laboratory flotation procedure.

Diagnostic testing should make extensive use of Six Sigma quality management principles such as "design of experiments." Because of the extensive number of factors that must be evaluated and the typical situation involving lack of enough material to perform a full factorial designed experiment, other types of two-level screening designs that can handle a large number of factors in a small number of tests are typically employed (Cytec 2010). Such designed tests provide valuable information on the main effects of variables (parameters) and serve to identify conditions for good performance and conditions to avoid. At least two replicates are recommended in these designed tests. The following are often the most commonly used parameters (there may be other factors that may be important in specific cases):

- Particle size distribution
- Desliming
- Ore type/mineralogy
- pH
- Collector type
- Collector dosage
- Collector addition point
- Frother type
- Frother dosage
- Frother addition point
- Modifier type
- Modifier dosage
- Modifier addition point
- Water chemistry
- Airflow rate
- Impeller speed
- Machine type
- Cell, impeller, and stator size
- Pulp level
- Pulp density in flotation

Many of these parameters may already be known, especially in the case of existing operation optimizations. Once the important parameters have been determined and a benchmark laboratory procedure has been established, the flotation conditions and reagents can be further optimized with a more focused laboratory test program. Appropriately designed experiments are required to generate statistically meaningful outcomes. During the design phase, the preliminary main and second-order interaction effects are determined with partial factorial experimental designs. Statistical analysis software packages (Minitab; JMP) are available to assist in the design and interpretation of results (Napier-Munn 2014; Cytec 2010).

Optimization Stage

The most impactful variables identified in the diagnostic-stage test work are the focus of the optimization stage. Further designed experiments using more extensive partial factorial or full factorial designed experiments with multiple levels are used to determine effects and nonlinear responses to process changes. This process is discussed further in other chapters of this handbook.

Rougher-Scavenger-Cleaner Laboratory Testing

Details of types of circuits investigated in the laboratory are given in Table 5. Rougher-scavenger, rougher-cleaner, and multistage flotation circuit designs are very common in non-sulfide systems. Their replication in the laboratory is not as simple as a rougher circuit alone, especially when the circuits involve multiple stages and presence of non-flotation separations such as magnetic or gravity separation. A guide on how to perform calculations commonly used in rougher-scavenger-cleaner flotation circuits can be found elsewhere in the literature (Kawatra 2011).

Bench-Scale Testing

Bench-scale testing methods (rougher, cleaner-recleaner, locked-cycle, kinetic) are described in Chapter 7.9, "Applied Flotation Modeling." Locked-cycle testing is a means of simulating plant conditions by recycling water and certain streams

Table 5 Laboratory investigated circuit types

Type of Circuit	Features and Use	Examples
Rougher-scavenger	• In direct flotation when rougher is capable of reaching the target grade, but the recovery is not optimal; the opposite in reverse flotation. • If the rougher float is a direct float, the tails (sinks) can require additional collector, frother, and modifier as well as further conditioning or grinding prior to scavenging. • If the rougher float is a reverse float, residual flotation reagents in the tails (floats) are, in many cases, sufficient for the scavenger float. • Maintain the pH between the rougher and scavenger. • Retaining the water from the rougher concentrate and using it in the scavenger float cell can give some indication to the effects of process water and residual reagents. • Size of the scavenger flotation cell can be reduced because of the low amount of solids in the rougher tails (feed to scavenger).	• Reverse flotation of silicate gangue from iron ores; rougher flotation with amine followed by single- or multistage scavenging
Rougher-cleaner	• Used when rougher is capable of reaching an acceptable recovery, but the grade is not optimal, because of complex ores or ores of wider size distributions. • If the rougher float is a reverse float, the concentrate (sinks) may require additional collector, frother, and modifier as well as further conditioning or grinding prior to scavenging. • If the rougher float is a direct float, residual flotation reagents in the concentrate (floats) are, in many cases, sufficient for the cleaner float. • Maintain the pH between the rougher and cleaner. • Retaining the water from the rougher tails and using it in the cleaner float cell can give some indication to the effects of process water and residual reagents.	• Barite flotation from oxide gangue; barite is floated using sulfate functionalized surfactants, and multiple cleaner floats are required to reach the desired grade.
Multistage flotation circuits	• Many flotation processes used for non-sulfide minerals are developed for complex ores in which a single flotation step may not be sufficient to provide the grade required. • Replication of a double-float or multistage flotation in the laboratory is more difficult. For example, de-oiling in the laboratory is typically performed by (1) conditioning the rougher concentrate at high solids in a separate cell with an acid, base, bleach, surfactant, or solvent; (2) allowing the solids a certain amount of time under agitation for the reagents to be scrubbed off the mineral surfaces; (3) washing over a wet sieve for a set amount of time with a set water flow rate to ensure reproducibility between tests; and (4) conditioning the scrubbed solids under new flotation conditions prior to reverse flotation. • Water in each stage will be segregated because different reagent schemes and pH are used.	• Double-float or Crago process (Crago 1942) for sedimentary phosphate ores; direct flotation of phosphate minerals with a fatty acid, followed by de-oiling of phosphate concentrate and reverse flotation of silica with an amine • Five-stage spodumene–mica–feldspar–quartz float previously used at Kings Mountain, North Carolina, United States (Redeker 1981; Kawatra and Carlson 2013)
Flow sheets with combination of flotation and non-flotation separation stages	• Most industrial flotation circuits have processing steps before or in between flotation stages. • Desliming is used with fine feeds to remove slime material prior to flotation. • Scrubbing is performed between flotation stages at some plants where multistage flotation is required. • Grinding can be performed between rougher and scavenger floats to liberate coarse middlings. • Size separation can be performed between flotation stages to remove coarse middlings particles via screens or cyclones.	• Magnetite beneficiation uses magnetic separators to reject liberated silicates prior to flotation. • Many carbonaceous phosphate ores are deslimed prior to flotation. • Gravity separation in many circuits (e.g., beryl, rare earths).

from test to test. For non-sulfide ores, this type of testing is especially important for ores containing minerals with appreciable solubility (e.g., carbonates, sulfates, phosphates). However, locked-cycle testing is cumbersome when the flow sheet involves multiple stages of flotation and/or non-flotation separations such as magnetic separation. Each stage may have to be isolated and subjected to locked-cycle testing. Pilot-plant testing is typically a far better option than locked-cycle testing for non-sulfide minerals, especially when complex flow sheets are involved.

PILOT-PLANT FLOTATION OF NON-SULFIDE MINERALS

Flow sheets for non-sulfide ore beneficiation tend to be more complex than those of sulfide ore systems, and simulating the whole flow sheet in batch laboratory flotation or locked-cycle testing is cumbersome. Pilot-plant testing is a far better option.

The sequence of testing is shown in Figure 3. If the ore sample or ore deposit under investigation is from, or similar to, an operating plant, then batch laboratory testing may be adequate, and pilot-plant testing can be bypassed. However, new flotation processes or those that have no close counterpart in current operating plants usually require piloting. A decision to have or not have a demonstration flotation plant should be based on the need to resolve potential process risks and/or unknowns in the proposed flow sheet, generate sufficient flotation concentrate to demonstrate downstream hydrometallurgical or pyrometallurgical processes, or evaluate mineral product end use.

Evaluation of Pilot-Plant Testing

The actual pilot-plant program is highly dependent on sample availability, mineralogy, metallurgical parameters, and flow-sheet considerations, as well as the budget.

Flotation Reagents for Pilot Testing

All reagents to be used during the pilot test should be prepared to the required strengths prior to commencement. Reagent preparation guidelines are available from the suppliers and in the published literature. Water-soluble reagents are added as aqueous solutions by metering pumps. For reagents that are insoluble or only sparingly soluble in water, special techniques are necessary, such as dissolving them in an organic solvent or emulsifying the reagent in water by mixing it with a suitable surfactant. In many instances, the insoluble reagent may exist as a liquid dispersion or a colloid. Accurate addition of reagents is necessary to quantify their effect on pilot metallurgical performance. With knowledge of feed rate of dry solids and the strength of the prepared reagents, the addition of the reagent can be calculated.

Equipment and Operations

Bench-scale parameters can be scaled up to the pilot scale, typically using a retention time required for the desired separation. The pilot-plant feed rates are usually guided by cell volumes and scaled retention times (Table 4). The slurry density guides the reagent addition rates. Typically, a flotation pilot plant is operated for several hours with minor reagent adjustments to obtain the steady-state conditions. The circuit is then operated for a few hours prior to sampling the circuit under these steady-state conditions. This usually lasts 8 hours to five days and is a continuous operation. Pilot flotation testing can be automated by using advanced control techniques to minimize experimental error.

Data Collection and Analysis

The pilot circuit is sampled over several hours to produce a composite sample of feed, concentrate, and tailings. Three to five composites are collected under steady-state conditions to

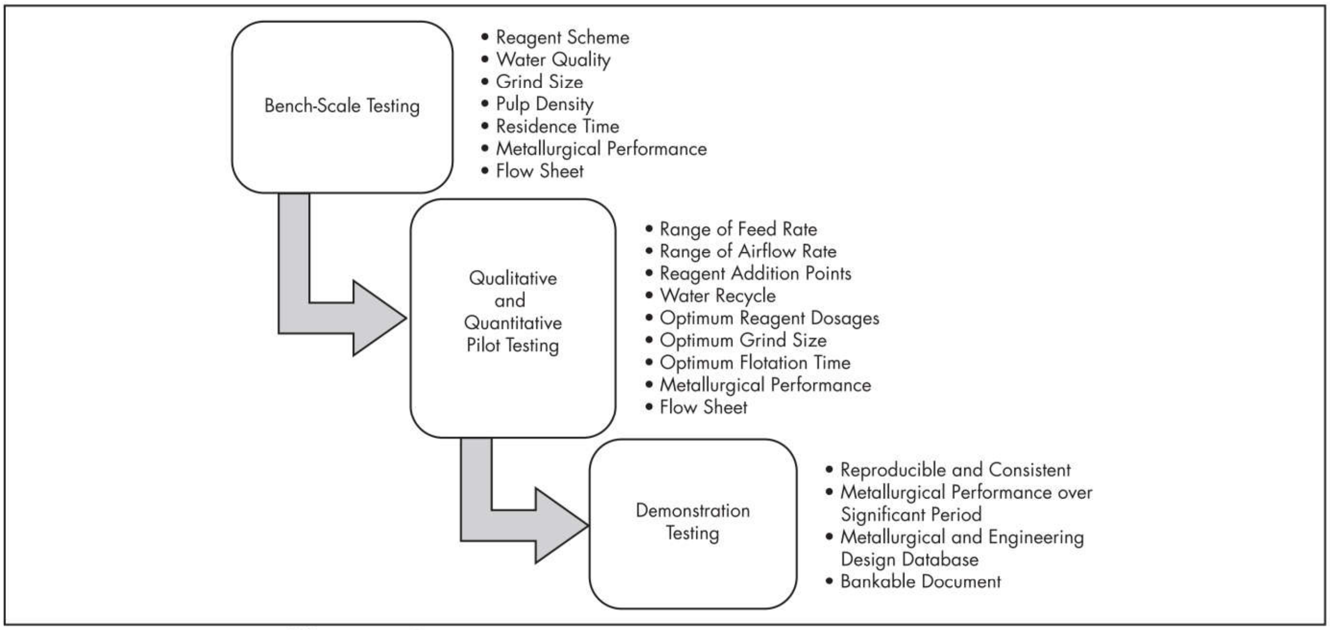

Figure 3 Key components of flotation pilot testing

Table 6 Summary of flotation development techniques

	Bench-Scale Testing	Locked-Cycle Testing	Pilot-Plant Testing
Objectives	• To evaluate major variables such as – Mineralogical variation – Head grade variation – Liberation and grind size requirements – Reagent types, quantities, conditioning time, and addition points – Concentrate regrinding requirements – Pulp density and viscosity effects – Water quality – Flow-sheet alternatives • To define the kinetics and separation response • To define valuable mineral recovery and grade of the saleable concentrate	• To determine the effect of recirculating material from cleaner circuit on recovery • To evaluate the variation in reagent dosages to compensate for the recycling of reagents, water, soluble metal species • To study the effect of the build-up of slimes or middlings in the cleaning stages of the test • To evaluate froth handling problems	• To confirm metallurgical response for continuous operation • To generate sufficient concentrate and tailings samples for downstream processes for end-use study • To obtain operating data for use in engineering design and scale-up purposes
Sample source	Existing operations, drill-core composite samples, variability samples	Existing operations, drill-core composite samples, variability samples	Bulk samples
Sample requirement	200–2,000 g per flotation test	10–50 kg per locked-cycle test	5–1,000 t
Flotation equipment	0.5–5 L mechanically agitated flotation machine	0.5–5 L mechanically agitated flotation machine	Forced air, self-aspirated, column, or shear contact pilot flotation cell banks
Limitations	• Does not allow metallurgical evaluation under conditions of continuous operation. • Does not allow evaluation of recycling of reagents or middlings. • The laboratory flotation feed preparation process (grinding and conditioning) produces different size distributions and chemical environments compared to an operating plant. • Difficult to simulate the whole flow sheet; difficult to simulate certain unit operations such as flash flotation, column flotation.	• Tests are time consuming, so it may not be possible to complete a test in one working day. • Requires rapid reporting of analytical results. • Aging of the intermediate products. • Cumbersome when the flow sheet is complex and contains non-flotation separations.	• Requires a large amount of representative and homogeneous feed sample. • High costs are involved in generating large quantities of sample for pilot-plant testing. • Requires storage and handling of tailings.

Note: Information in this table is from Barbery et al. 1986; Cameron and Dunlop 1990; Cytec 2010; Gochin and Smith 1987; Macdonald and Brison 1962; Williams et al. 2002; Wills and Napier-Munn 2006; and authors' experience.

estimate the standard deviation in the results. The data collected from a pilot-plant testing can be considerable, ranging from mass and water recoveries, to assay and mineralogical data, to pulp chemistry measurements. For pilot-plant control, pulp densities, plant feed rate, feed sizing, and reagent flows are regularly monitored. Concurrent with the pilot sampling, additional bench flotation tests may be performed on samples taken at the specified sample points in the flow sheet as a control; this is considered best practice.

Reporting

A typical pilot-plant report contains a comprehensive analysis and interpretation on circuits most applicable for treatment of the ore body, some design data and marketable grades, and recoveries possible. If required, the report can be produced to bankable document standards and include engineering design data (Cameron and Dunlop 1990; Williams et al. 2002).

SUMMARY

There are many factors to consider when developing flotation technology for non-sulfide minerals. A summary of the different techniques used for non-sulfide flotation development is shown in Table 6. Generally, the steps required to design a flotation circuit for non-sulfide minerals are as follows:

1. Sampling of the feed from drill cores or an existing operation
2. Mineralogical characterization of the feed
3. Liberation analysis of the feed
4. Grinding study if not adequately liberated
5. Bench-scale flotation testing
6. Locked-cycle testing
7. Pilot-plant testing
8. Full plant design implementation and evaluation

REFERENCES

Aplan, F.F. 1994. Reagents for the flotation of non-sulfide ores In *Reagents for Better Metallurgy*. Edited by P.S. Mulukutla. Littleton, CO: SME. pp. 103–117.

Aplan, F.F., and Fuerstenau, D.W. 1962. Principles of nonmetallic mineral flotation. In *Froth Flotation: 50th Anniversary Volume*. Edited by D.W. Fuerstenau. New York: AIME. pp. 170–214.

Arbiter, N., and Williams, E.K.C. 1980. Conditioning in oleic acid flotation. In *Fine Particles Processing: Proceedings of the International Symposium on Fine Particles Processing*. Edited by P. Somasundaran. Littleton, CO: SME-AIME. pp. 802–831.

Baarson, R.E., Ray, C.L., and Treweek, H.B. 1962. Plant practice in nonmetallic mineral flotation. In *Froth Flotation: 50th Anniversary Volume*. Edited by D.W. Fuerstenau. New York: AIME. pp. 427–454.

Barbery, G., Bourassa, M., and Maachar, A. 1986. Laboratory testing for flotation circuit design. In *Design and Installation of Concentration and Dewatering Circuits*. Edited by A.L. Mular and M.A. Anderson. Littleton, CO: SME. pp. 419–432.

Cameron, P., and Dunlop, G. 1990. Pilot plant testing in mineral process plant design. In *Mining Industry Capital and Operating Cost Estimation Conference*. Melbourne, Victoria: Australasian Institute of Mining and Metallurgy. pp. 29–34.

Coghill, W.H., and Clemmer, J.B. 1935. Soap flotation of the nonsulfides. *Trans. AIME* 449–465.

Crago, A. 1942. Process of concentrating phosphate minerals. U.S. Patent 2,293,640.

Cytec. 2010. *Mining Chemicals Handbook*. Woodland Park, NJ: Cytec Industries.

Davis, F.T. 1985. Nonmetallic industrial minerals. In *SME Mineral Processing Handbook*. Edited by N.L. Weiss. Littleton, CO: SME-AIME.

Dunne, R.C., Lane, G.S., Richmond, G.D., and Dioses, J. 2013. Flotation data for the design of process plants: Part 1—testing and design procedures. *Miner. Process. Extr. Metall.* 199–204.

Falconer, S.A. 1961. Minerals beneficiation—Non-sulfide flotation with fatty acid and petroleum sulfonate promoters. *Trans. AIME* 217:207–215.

Falconer, S.A., and Crawford, B.D. 1947. Froth flotation of some nonsulphide minerals of strategic importance. *Trans. AIME* 527–542.

Fuerstenau, D.W., ed. 1962. *Froth Flotation: 50th Anniversary Volume*. New York: AIME.

Fuerstenau, D.W. 2007. A century of developments in the chemistry of flotation processing. In *Froth Flotation: A Century of Innovation*. Edited by M.C. Fuerstenau, G. Jameson, and R-H. Yoon. Littleton, CO: SME. pp. 3–64.

Fuerstenau, M.C., Jameson, G., and Yoon, R.-H. 2007. *Froth Flotation: A Century of Innovation.* Littleton, CO: SME.

Gaudin, A.M., and Glover, H. 1928. Flotation of some oxide and silicate minerals. Technical Paper No. 1. Salt Lake City: Department Mining and Metallurgical Research, University of Utah. pp. 78–101.

Gochin R.J., Smith M.R. 1987. The methodology of froth flotation testwork. In *NATO Science Series E: Applied Sciences.* Vol. 122, Mineral Processing Design. Edited by B. Yarar and Z.M. Dogan. Dordrecht, Germany: Springer.

Holme, R.N. 1986. Sulfonate-type flotation reagents. In *Chemical Reagents in the Mineral Processing Industry*. Edited by D. Malhotra and W. Riggs. Littleton, CO: SME. pp. 99–111.

Houot, R. 1983. Beneficiation of iron ore by flotation: Review of industrial and potential applications. *Int. J. Miner. Process.* 10:183–204.

Jones, M.J., and Oblatt, R., eds. 1984. *Reagents in the Minerals Industry*. London: Institution of Mining and Metallurgy.

Kawatra, S.K. 2011. Fundamental principles of froth flotation. In *SME Mining Engineering Handbook*, 3rd ed. Edited by P. Darling. Englewood, CO: SME. pp. 1517–1531.

Kawatra, S.K., and Carlson, J.T. 2013. *Beneficiation of Phosphate Ore*. Englewood, CO: SME.

Macdonald, R., and Brison, R. 1962. Applied research in flotation. In *Froth Flotation: 50th Anniversary Volume*. Edited by D.W. Fuerstenau. New York: AIME. pp. 298–327.

Malhotra, D., and Riggs, W.F. 1986. *Chemical Reagents in the Mineral Processing Industry*. Littleton, CO: SME.

Manser, R.M. 1975. *Handbook of Silica Flotation*. United Kingdom: DOB Services (Hitchin).

Miller, J.D., Tippin, B., and Pruett, R. 2002. Nonsulfide flotation technology and plant practice. In *Mineral Processing Plant Design, Practice, and Control*. Edited by A.L. Mular, D.N. Halbe, and D.J. Barratt. Littleton, CO: SME. pp. 1159–1178.

Miller, J.D, Abdel Khalek, N., Basilio, C., El-Shall, K., Fa, K., Forssberg, K.S.E., Fuerstenau, M.C., Mathur, S., Nalaskowski, J., Rao, K.H., Somasundaran, P., Wang, X., and Zhang, P. 2007. Flotation chemistry and technology of nonsulfide minerals. In *Froth Flotation: A Century of Innovation*. Edited by M.C. Fuerstenau, G. Jameson, and R-H. Yoon. Littleton, CO: SME. pp. 465–553.

Mulukutla, P.S., ed. 1994. *Reagents for Better Metallurgy*. Littleton, CO: SME.

Nagaraj, D., and Farinato, R. 2016. Evolution of flotation chemistry and chemicals: A century of innovations and the lingering challenges. *Miner. Eng.* 96-97:2–14.

Nagaraj, D., and Ravishankar, S. 2007. Flotation reagents—A critical overview from an industry perspective. In *Froth Flotation: A Century of Innovation*. Edited by M.C. Fuerstenau, G. Jameson, and R-H. Yoon. Littleton, CO: SME.

Napier-Munn, T. 2014. *Statistical Methods for Mineral Engineers: How to Design Experiments and Analyse Data*. Indooroopilly, Queensland: Julius Kruttschnitt Mineral Research Centre.

NC State University. 2018. Minerals Research Laboratory: Minerals research lab reports. https://mrl.ies.ncsu.edu/minerals-research-lab-reports/. Accessed August 2018.

Peres, A.E.C., Araujo, A.C., El-Shall, H., Abdel-Khalek, N.A. 2007. Plant practice: Nonsulfide minerals. In *Froth Flotation: A Century of Innovation*. Edited by M.C. Fuerstenau, G. Jameson, and R-H. Yoon. Littleton, CO: SME.

Redeker, I. 1981. Flotation of feldspar, spodumene, quartz and mica from pegmatites in North Carolina, USA. CMP Preprint No. 81-1-P. Ottawa, ON: Canadian Mineral Processors.

Redeker, I., and Bentzen, E. 1986. Plant and laboratory practice in nonmetallic mineral flotation. In *Chemical Reagents in the Mineral Processing Industry*. Edited by D. Malhotra and W. Riggs. Littleton, CO: SME. pp. 3–20.

Siirak, J., and Hancock, B.A. 1988. Progress in developing a flotation phosphorous reduction process at the Tilden iron ore mine. In *XVI International Mineral Processing Congress*. Amsterdam: Elsevier. pp. 1393–1404.

Somasundaran, P., and Moudgil, B.M., eds. 1987. *Reagents in Mineral Technology*. Surfactant Science Series 27. New York: Taylor and Francis.

Spedden, H.R. 1985. Sampling and testing. In *SME Mineral Processing Handbook*. Edited by N.L. Weiss. Littleton, CO: SME-AIME.

Williams, S., Ounpuu, M., and Sarbutt, K. 2002. *Bench and Pilot Plant Programs for Flotation Circuit Design*. Technical Paper No. 2002-04. Rutherford, NJ: SGS Mineral Services.

Wills, B.A., and Napier-Munn, T.J. 2006. Froth flotation. In *Wills' Mineral Processing Technology: An Introduction to the Practical Aspects of Ore Treatment*, 7th ed. Oxford: Elsevier Science and Technology Books. pp. 267–352.