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Fine Particle Separation by Selective Flocculation*

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Abstract

One of the most promising techniques for separating very small particulates is selective flocculation. A number of successful applications for selective flocculation have been demonstrated on laboratory and pilot-plant testing, but with only a few commercial applications. Understanding of the process parameters and potential applications is still at the early stages, and research is underway to develop this process for cooper, coal, phosphate, and tin ores. Thus far, most of the applications of selective flocculation have been in minerals processing, where separation of the valuable minerals from associated waste rocks is the objective. But potential applications for selective flocculation also exist in other areas, such as the removal of heavy metal oxides from steel dusts and other hazardous incineration residues, separation of the impurities from ceramic mineral powders, the removal of opaque minerals from kaolin, and the recovery of values from secondary sources. In this article the methods involved in obtaining selective flocculation, examples of successful applications, recent developments, and future outlook for this technology are discussed.

INTRODUCTION

One of the most promising techiques for separating very small particles is selective flocculation. This technique utilizes the differences in the physical-chemical properties of various types of particles in the mixed suspension. The technique is essentially based on the preferential absorption of an organic flocculant on the particular colloidal solids to be flocculated, leaving the remainder of the particles suspended. Considerable success has been achieved in applying this technique for the recovery of finely disseminated valuable minerals from a number of natural ores on bench-scale, pilot-plant, and commercial operations (*1–15*). Research is already under way to

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develop and demonstrate the applicability of this technique to several other ore types such as tin, copper, coal, and phosphates.

However, in order that full potentials of this emerging technology could be realized in a short time, understanding of the scientific principles of selective flocculation must keep pace. Much progress has already been made in delineating these principles (*4*, *5*), but understanding the basis of designing the process parameters is far from complete.

At the outset, there are three main methods for achieving aggregation of very small or colloidal particles in suspension that are often confused with each other. In one method, aggregation is caused by the compression of the electrical double layer of the solids by the addition of simple electrolytes. This method is known as *coagulation* and the aggregates are known as coagula. The second method of aggregation is through the use of long-chain organic polymers that bridge between the adjoining particles. This method is known as *flocculation* and the aggregates are known as flocs. In the third method where aggregation is caused by the action of hydrolyzing electrolytes, such as ferric or aluminum sulfate, the mechanism involved is a composite of both coagulation by the polyvalent ions and bridging flocculation by the precipiting hydroxides. All of these methods have been used in water and waste treatment where total flocculation of these solids is the objective. Thus far, only the first two methods could be used to achieve aggregation of certain types of particles in a mixed suspension.

Selective coagulation, like selective flocculation, must start with an initially deflocculated slurry, but unlike selective flocculation it is based on exploiting the differences in the slow coagulation rates of the various constituents (*6*). All of the particles, however, must carry the same sign of surface charge in order to avoid rapid mutual coagulation.

Other methods of aggregating fine particles from suspensions, such as oil agglomeration, where oil droplets provide the bridging bonds between the adjoining particles, can be used for selective agglomeration of hydrophobic solids from mixed aqueous suspension with hydrophilic particles. Discussion of this technique, however, is outside the scope of this article.

Of all these methods, selective flocculation by polymers appears to offer the most promise for the commercial separation of very small particles such as in the recovery of fine valuable minerals from natural ores and tailings as well as in other nonmineral systems. Examples of commercial applications of selective flocculation for iron ores and potash are discussed elsewhere (*2*, *3*).

The aim of this article is to discuss briefly the methods involved in obtaining selective flocculation, recent development, and future outlook for this technology.

DESIGN STRATEGY OF THE SELECTIVE FLOCCULATION PROCESS

In designing a selective flocculation process to separate certain desired particles from mixed suspension with unwanted (gangue) solids, selective flocculation of either the valuable components or the gangue components may be employed depending on which route is more technically and economically viable. Both of these routes have been successfully applied on a commercial scale. In one application for processing iron ore (*2*), the valuable components, iron minerals, were selectively flocculated while the associated gangue minerals (silica and silicates) remained dispersed. The iron mineral flocs were separated from the suspension by gravity settling using a thickener. In the other application processing potash ore (*3*), the unwanted minerals (clays) were selectively flocculated while the halide minerals remained dispersed. The clay flocs were separated from the suspension by flotation of the flocs. Thus, in principle, the separation of certain fine particles from mixed suspensions could be obtained by either route.

There are some general guidelines to be followed which are similar to those used in the application of froth flotation. More specifically to selective flocculation, the choice of either route would be influenced by the available knowledge of the surface and flocculation behaviors of each of the components under various environments of reagents suite. Generally, the route that would result in a lower reagent consumption, more efficient separation, and lower equipment cost (both capital and operating cost) would obviously be the preferred choice.

However, this decision is usually reached after a considerable amount of work on bench and pilot-plant scale. In setting out to design a selective flocculation process from fundamental or basic levels, knowledge of the various methods of achieving selective flocculation is essential.

METHODS OF ACHIEVING SELECTIVE FLOCCULATION

There are basically three ways to achieve selective flocculation of the mineral of choice (whether it be the valuable or the unwanted minerals) from a mixed suspension. These are:

I. Use of a selective flocculant for the minerals of choice, either alone or in combination with a general, unselective dispersant
II. Use of a selective dispersant in combination with a normally unselective flocculant

III. Use of a selective activator in combination with a general but nonadsorbing flocculant.

Obviously, the use of any combination of these methods would be expected to achieve superior performance to any of the methods alone. However, this might not necessarily be the most cost effective, since the cost of preparing selective reagents might be inherently more expensive, at least at this early stage of development, than the more commercially produced reagents.

I. Use of a Selective Flocculant

This route involves employing a selective flocculant either alone or in combination with general dispersants. Obtaining a selective flocculant absorption could be made by either of the following means:

A. Designing a specific flocculant for the particular mineral (solids) surface, ideally achieving a yes or no adsorption. These specific polymeric flocculants can be classified into the following categories:

Chemisorbing or chelating polymers in which the functional groups are strong complex formers with certain ions on the solid surface.

Stereoselective flocculants which rely on the selective effect of the molecular structure such as steric hinderance of adsorption of polymers having bulky constituent groups in their structures.

Hydrophobic or partially hydrophobic flocculants where selective flocculation of hydrophobic minerals (or other hydrophobic solids) from those hydrophilic ones are desired.

B. Enhancing the polymer's selectivity by the chemical modulation of interfaces, which could be implemented by regulating the surface electrical potential or by employing competitive dispersion conditions. In the latter, the dispersant or depressant would compete with the flocculant for bonding sites on a certain solid surface, thereby inhibiting its adsorption. This might also be considered as a case of selective dispersion.

The design of selective flocculation processes using selective flocculants by one or more of these methods has been the major route followed by researchers thus far. Examples of selective chemisorbing flocculants are found in the literature (5). Notably, the development of polyxanthate flocculants for the selective flocculation of sulfide minerals and oxidized copper minerals was first reported by Attia and Kitchener (*4*), and was later confirmed by Sresty et al. (*7*) and Baudet et al. (*8*). Other chelating polymers were designed and used for the selective flocculation of copper minerals from a natural ore (*9*) and for cassiterite separation from quartz (*10*). The use of chemisorbing or chelating polymers for achieving selective flocculation so far represents the most practical and promising method. These types of flocculants are, therefore, very likely to find wide applications in the mineral processing industry in the future.

Stereoselective flocculants have not yet been designed as such or used in selective flocculation although their potential has been recognized (*11*) for some time. The design of this type of flocculants involves the geometrical arrangement of the flocculant's functional groups, so as to match the geometrical structure of the binding ionic (or atomic) sites on certain solid surfaces, thereby selectively adsorbing and flocculating them. Structural effects, such as steric-hinderance, ring strain, and specific size fit (of a chelating flocculant), could be employed to produce selective flocculation. Future developments in flocculant design will likely take advantage of this rather sophisticated concept.

Hydrophobic or partially hydrophobic flocculants appear to have the greatest potential for selectively flocculating hydrophobic solids (with natural or induced hydrophobicity) in mixed suspensions with hydrophilic solids. The use of this type of flocculants for selectively flocculating *naturally* hydrophobic solids, such as coal, was first suggested by Attia (*12*). However, the application of this type of flocculant in the selective flocculation of hydrophobized copper minerals was first reported by Rubio and Kitchener (*13*). In their application, the initially hydrophilic surfaces of oxidized copper minerals were selectively made hydrophobic by selective adsorption of surfactants or even by sulfidization with sodium sulfide. Then by introducing the partially hydrophobic flocculant, polyethylene oxide, selective flocculation of oxidized copper minerals from associated hydrophilic silica and silicate minerals was achieved.

Obtaining selective flocculant adsorption through the control and modulation of interfaces was probably the only route for achieving selective flocculation before the discovery and development of new, more selective flocculants. Regulation of surface potential for achieving selective flocculation is expected to act in at least two modes; (a) control of the degree of adsorption of the charged, ion-containing flocculants, and (b) control of the degree of repulsion between similarly charged particles within the floc. A high degree of repulsion between the particles could result in weakening of the floc strength or even inhibiting floc formation. Neutral or dissimilarly charged particles could enhance flocculation by undergoing slow or fast "coagulation" depending on the magnitude of the operating attractive forces.

The use of surface potential regulation or competitive dispersion conditions to achieve selective flocculation is usually accomplished in actual practice by the adjustment of suspension pH and by the addition of various dispersants. The combined effect of pH and dispersants is usually tested for a wide range of flocculants in order to find the right conditions for achieving selective flocculation. The literature contains numerous examples of employing chemical modulation of interface to achieve selective flocculation. One such example reported by Friend and Kitchener (*14*) was the separation of calcite from mixtures with rutile by an ionic flocculant, the calcite being the flocculated component. Values of zeta-potential were controlled by adjustment of pH and sodium tripolyphosphate (STPP) concentration.

II. Use of Selective Dispersants

The primary function of a dispersant is to reduce the gravitational settling and the aggregation rate of the individual particles ideally to very low values or zero. The dispersing agent could be either monomeric or polymeric in nature; the latter could either be linear or branched molecules. Since more surface coverage is usually required in the case of dispersion compared with the flocculation, branched molecules should be more effective than linear ones. On the contrary, the effective flocculant is normally a long-chain, linear molecule which is needed for bridging between the adjoining particles in the floc.

In general, dispersants for aqueous solid suspensions may function by one or more of the following mechanisms:

(1) Control of surface electrical potential in order to induce repulsion between similarly charged particles
(2) Reduction of interfacial energy of solids by surface-active dispersants
(3) Solvation of particle surface by the adsorption of hydrophilic colloids

In the last mechanism the particle–particle interaction would be reduced to liquid–liquid interaction due to the presence of lyophilic layers on the particle surface. It is difficult to single out one mechanism that is the most important for selective dispersion-flocculation, but it is likely that the first mechanism will be the most useful.

In addition to the dispersant function, the same reagent must also act as a depressant for the flocculant action on the solid particles by competing with it for bonding sites on the surface and thus inhibiting its adsorption. The "dispersant" is also often required to act as a deactivating agent by complexing potential interferring ionic species present in the suspension.

However, in order for the reagent to perform these dispersion and depression functions, it must first adsorb on the solid particles. The adsorption mechanisms are very similar to those for flocculant adsorption. Therefore, the same guidelines for designing selective flocculants should also apply to developing selective dispersants. Namely, the design of selective chemisorbing dispersants, partially hydrophobic, and stereoselective dispersants should be possible. These dispersants when used with a general type flocculant would allow all the suspension particles to be flocculated, except those ones which they have adsorbed on.

As with flocculants, attention should be paid to the solution chemistry of the dispersants as it relates to their actions in the suspension. For instance, the effect of suspension pH, ionic strength, and other interactive ionic species present should be identified and checked. Further, it is often very beneficial to employ more than one type of dispersant simultaneously instead of just one. The combined effect of multiple dispersants are often much greater than either of the individual dispersant alone. Presumably, one type of dispersant complements the other with regards to fulfilling the various dispersion, depression, and ionic deactivation functions required in the system.

The possibility of designing selective chemisorbing dispersants was confirmed by Attia (*1*), who found that by using modified low molecular weight polymers with chemical groups that are selective toward certain solid surfaces in the mixed suspension, selective dispersion and depression could be obtained. Thus the author designed and prepared two novel reagents for magnesium and calcium-bearing minerals, such as calcite and dolomite. The crude solution of one reagent tested exhibited selective dispersant and depressant behavior for calcite and dolomite but to a much lesser degree for feldspar. Lower concentrations of this reagent, compared with the unmodified polymer, enabled more effective selective flocculation of the copper minerals from mixtures with calcite, dolomite, and feldspar. This reagent also proved to be a selective depressant for calcite and fluorite in their separation from barite by flotation.

There are a number of commercial reagents that exhibit some degree of selectivity toward various mineral surfaces. For example, sulfonated polystyrene was used for the selective dispersion of clay minerals while flocculating the coal particles with a polyacrylamide flocculant (*15*). In addition, there are a number of commerically available reagents that are capable of selectively adsorbing onto hydrophobic surfaces and, therefore, achieving selective dispersion or depression. Future developments will no doubt make use of such reagents in processing hydrophobic surfaces, such as coal and talc, by selective flocculation technology.

III. Use of Selective Activators

By analogy with the use of selective dispersants or depressants, use of selective activators might also be used to achieve selective flocculation. The use of selective activators for certain components of the mixed suspension which, when used with a general but nonadsorbing flocculant (for example, a highly anionic flocculant at high pH), would cause the selective flocculation of these components. The choice and design of the selective activating agents would be governed by the same principles used for selective flocculants or dispersants.

FUTURE OUTLOOK FOR SELECTIVE FLOCCULATION TECHNOLOGY

Although the technology has found some successful applications in the mineral processing industry, future research developments will focus on (a) finding new applications, (b) developing the process parameters, (c) designing of new chemical reagents, and (d) designing of appropriate equipment.

Many applications of selective flocculation have already been achieved on a bench-scale testing or pilot-plant operation, with only two major commercial applications known. Future developments will witness commercialization of this technology to a wide variety of ore types.

It is also anticipated that the application of selective flocculation will extend beyond mineral processing into areas such as the removal of heavy metal oxides from steel dusts and other hazardous incineration residues, separation of the impurities from ceramic mineral powders, the removal of opaque minerals from kaolin, the recovery of values from secondary sources, and other applications where very small particles are involved.

In order for these applications to be successful, understanding of the process parameters in a more quantitative fashion as well as development of new reagents and appropriate equipment should be made. The process parameters are currently being examined by the author, and the findings of this and other studies should constitute a major step forward for firmly establishing this separation technology.

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