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Chapter 14

PRINCIPLES OF SELECTIVE AGGREGATION

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INTRODUCTION

Large amounts of mineral values are discarded today as fines and ultrafines because of inadequate technology to process them economically. Most conventional mineral processing techniques fail in the sub-sieve range. Fines respond poorly to processes such as flotation either due to change in mineralogy and surface composition or even simply due to the direct effect of their smallness in size on collision and adhesion rates between them and air bubbles. Indeed if the poor flotation is due to the smallness in size, a solution must rest in aggregating the fines. Inorganic electrolytes as well as polymers have been successfully used as flocculants in the past for obtaining increased flotation of colloidal material from waste water effluents. In mineral beneficiation, this technique can be profitably used if the process of flocculation can be made to take place selectively. The power of the selective flocculation technique has already been proven, with the successful application of it in the case of beneficiation of low grade iron ore by the Cleveland-Cliffs Iron Ore Company and potash ore by Cominco. The process holds tremendous potential when it is followed by flotation, elutriation, etc. Today its application is, however, limited to the above two cases and it has become clear that a full understanding of the process will prove helpful for developing solutions to various problems that exist both at the basic and applied levels. In this paper, principles that are known to govern selective aggregation processes such as selective flocculation, carrier flotation and spherical agglomeration will be examined and areas of uncertainties will be identified.

BASIC PRINCIPLES

The first requirement for selective aggregation* is the complete dispersion of particulate components and then it is necessary to induce aggregation among the particles of one or more desired components leaving all the other components in a dispersed state. One can formulate physico-chemical conditions that will produce such selective aggregation by examining the nature of different interactions that exist between the particles in solutions. The interactions to be considered are:

a) collisions and forces governing the collision rate;
b) adhesion during collision and repulsive and attractive forces controlling the probability of adhesion, and
c) effect of dissolved mineral ions and other ions in water on surface properties of individual minerals and selectivity.

a) Collisions

Colloidal particles will undergo collisions, actual or aborted, due to thermal motions that are determined by particle size, temperature, viscosity, etc. Both collision rate and adhesion rate can be enhanced by controlled agitation by stirring. Flocculation assisted by such external forces is known as orthokinetic flocculation. Under turbulent conditions, collisions take place, either due to diffusion while entrained in eddies or due to inertia and the rate for a system containing n_i particles initially is given, according to Levich (2),as

$$J_{turb} = 12\pi\beta r^3 n_i^2 \left(\frac{\varepsilon_o}{\nu}\right)^{\frac{1}{2}}$$

where ε_o is the energy loss per unit volume per second, ν is the kinematic viscosity, r the radius and β a constant. The above expression can be used to calculate collision rate between particles of various sizes. By determining aggregation rate between such particles experimentally, adhesion rate can be calculated. Aggregation rates between fines and fines have been compared in this manner in one study to that between fines and coarse particles for one system and it was inferred that the probability of adhesion is larger for the latter case than for the former (3). Relevance of this observation to carrier flotation (ultraflotation) should be noted. It should be important to study this phenomenon further, particularly with a few other systems and to ascertain the general relevance of it. Also,

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*Following the IUPAC guidelines (1), the term aggregation is used here for the process or the result of the formation of a group of particles held together in any way. The formation of aggregates is called coagulation, if the aggregates are compact and flocculation if they are loose or open. Usually a coagulum is obtained with the addition of appropriate inorganic electrolytes to a sol and flocs are obtained with the help of polymers.

addition of carrier cannot be considered to simply enhance only the speed of the ultraflotation. It appears that it is in fact essential to achieve the required selectivity. Evidently, detailed work on the role of various parameters in orthokinetic flocculation is essential to make full use of it for treatment of fines.

Adhesion

Successful completion of the collision as well as adhesion of a permanent nature will depend upon the type of interactions between the particles. Interactions that are normally considered influential arise from London-van der Waals attractive forces, V_A, double layer forces that can be attractive or repulsive in nature, V_{el}, and steric forces that arise from the overlap of adsorbed layers, V_S. Steric forces can also be repulsive or attractive, depending upon whether the adsorbed layers will prefer to be in contact with the solvent or not. The nature of the sum total of the above three interactions, V_T, will determine whether a collision will be aborted or, if it takes place, whether the adhesion will be permanent. Models based upon DLVO and HHF theories (4) take into consideration the London-van der Waals attractive energy and electrical double layer energy and yield an expression for the total energy V_T which shows that it will be attractive at large and small distances. While the above considerations might be satisfactory for studying coagulation, it is of very little value for flocculation since in this case the interaction between polymer layers adsorbed on the two particles as well as that between such a layer and any bare particle surface will play a predominating role. To date, however, there are no fully developed models taking all the three energies into consideration. V_S can be positive or negative depending on the solvent power of the medium for the exposed portions of the adsorbed layer. If the solvent power for the exposed polymer branches is negligible, conditions will be favorable for interpenetration of the branches of the polymer adsorbed on different particles. Such interpenetration of the adsorbed layers will be naturally conducive to flocculation. It is also important to note that polymer adsorption in any significant amount can practically eliminate the influence of the original electrical double layer. In fact, we have observed the zeta potential of originally positive and negative hematite surfaces to be replaced by a single negative value upon adsorption of a polystyrene sulfonate (5). These considerations give the following criteria for selective aggregation between, say particles of component 1 from a mixture of 1 and 2.

1. Particles of both components 1 and 2 should preferably carry the same electrical charge so that there will be no aggregation between them. The magnitude of the charges should be such that the repulsive energy will be larger than the attractive energy. For this it is estimated that the zeta potential should be at least greater than about 20 mV in magnitude.

2. Charges on the particles to be aggregated, say of component 1, should be such that the repulsive energy between them will be less than that of the attractive energy. For this, zeta potential of these particles should preferably be, at least after the adsorption of the polymer, less than about 20 mV in magnitude.

3. Polymer should selectively adsorb on particles to be aggregated and should have limited solubility in the solvent. In some cases it will be sufficient to have considerable differences in the rate of adsorption of the polymer on the two components. Here, the differences in kinetics can be profitably used for obtaining selective flocculation.

According to the above, selective aggregation of mineral fines, of say component 1 from a mixture of components of 1 and 2, should in principle be possible by controlling the ionic composition of the solution so that $|\zeta_1| < -20$ mV, $|\zeta_2| > -20$ mV, and $\zeta_1|\zeta_2$ is positive. In practice, however, as we shall see later, such aggregation is rarely obtained. Why this is so is still a subject of speculation. In any case, it does become necessary to use polymers to obtain the required selectivity. The principles governing the adsorption of polymers and related kinetics are therefore, of primary importance in selective flocculation.

Selective Adsorption of Polymers and Flocculation. Adsorption of polymers on minerals is dependent on the polymer properties such as molecular weight and configuration, distribution of functional groups and effect of it on the configuration; mineral properties such as surface charge and oxidation state and solution properties such as ionic strength, temperature and solvent power for the polymer. It is this dependence of adsorption that can effectively be used for obtaining selective adsorption.

Forces responsible for polymer adsorption arise from electrostatic bonding, hydrogen bonding and covalent bonding.

Electrostatic forces play a governing role in the case of polymers with a large number of charged functional groups. For example, a cationic polymer, Nalcolyte-610, was found to be able to flocculate a negatively charged silica sample, (Biosil-A) while no detectable flocculation was obtained with Separan AP-30, which is anionic (6). Electrostatic adsorption of polymers can even lead to charge reversal of the particles, with the values obtained tending towards that expected for a monolayer of the polymer (5). However, it is to be noted that for flocculation to be effective, zeta potential of the particles need not be zero.

Adsorption of non-ionic type polymers by hydrogen bonding with, for example, surface oxygen groups of the mineral is not usually as selective as that obtained due to covalent bonding or electrostatic bonding.

Covalent bonding can be obtained between the active groups of the polymer and the cations of the mineral surface and this can usually provide the required selectivity in adsorption and resultant flocculation. Most long chain polymers are bulk flocculants with insufficient selectivity due to the absence of such groups. Selectivity can be induced in such cases either by altering the surface potentials of the mineral particles to create the desired electrical interactions between the ionized polymer chains and the mineral surfaces or by incorporating functional groups into the polymer.

Success of surface active agents as flotation collectors is known to depend on the presence of active groups such as carboxylate, sulfonate, mercaptan, amine, etc. in them. One way to obtain selectivity during flocculation should be by incorporating such groups that have been already known to cause adsorption on specific minerals and, clearly, work in this direction should prove very fruitful.

ACTIVATORS AND DEPRESSANTS: Again like in the case of flotation, it should be possible to enhance or retard selective flocculation by means of chemical additives that can adsorb on the mineral particles and activate or depress them. Thus separation of hematite-quartz mixture using anionic polyacrylamide has been reported to be promoted by the addition of Calgon and sodium fluoride (7). On the other hand, flocculation of heavy minerals by xanthates has been suggested to be depressed by the addition of sodium sulfide, polyphosphates and polyacrylates (8). Apart from such scant reports there has been very little work, however, on the role of these additives in obtaining selective flocculation.

KINETICS OF ADSORPTION: Some recent results suggest that selectivity can also be induced by making use of the differences in the rate of adsorption of the polymer on the minerals (6). The kinetics is dependent on the rate of diffusion of these molecules to the surface of the particles and are, therefore, influenced by concentration of the polymer in solution, as well as its molecular weight and configuration, temperature, agitation, and the mineral surface properties (9). These factors need not, however, affect the relative rates of adsorption on the minerals. The factors that might influence the relative rates are surface area and porosity, surface hydration, zeta potential, etc. Whether due to differences in the rate of adsorption or in rates of some other process controlling flocculation, significant differences in optimum reagentizing times have been observed for hematite and quartz using Separan AP-30 (6). The reasons for these differences are not, however, known at this point. Maximum flocculation was observed for hematite with about 60 seconds reagentizing around neutral pH whereas for quartz the corresponding time was 600 seconds. Prolonged reagentizing always caused redispersion of the flocculated mass possibly due to complete surface coverage of the particles by polymer. Such coverages have been reported to retard interparticle bridging and thus, to induce stabilization of the suspension (10,11). Partial

coverage allows the free loops of the polymer adsorbed on one particle to adsorb on the bare regions of another particle and thus produce interparticle bridging and thereby flocculation. Stability of suspension due to complete coverage of the particles by adsorbed polymer molecules is called steric stabilization and can be avoided only if the net change in Gibbs free energy due to interpenetration of the polymer chains is negative (12). For flocculation to proceed in such cases, the increase in entropy due to release of solvent molecules should outweigh the loss of entropy due to interpenetration of polymeric chains and increase in enthalpy due to increased degrees of freedom attained by the released solvent molecules. The relevance of this to the mineral systems are, however, yet to be ascertained.

Clearly information on the configuration of the polymer species adsorbed on mineral species under various conditions will prove helpful in this regard. Currently there is hardly any such information available for mineral systems.

Effect of Dissolved Ions

Often predictions of selective flocculation on the basis of results from single mineral tests do not agree with those obtained for natural ores containing these minerals and sometimes not even with those obtained for their synthetic mixtures. The problem is of a much more general nature since similar observations have been made during flotation studies also. A major factor for this might be the interference by the dissolved chemical species of one particle on account of either its adsorption or precipitation on the others. The above discrepancy is not completely understood. The role of various possible factors and particularly that of the presence of undesirable chemical species such as calcium and the sequestering agents that are used to counteract, needs careful and systematic study.

SELECTIVE AGGREGATION

Selective aggregation has been successfully accomplished in a number of ways and these have been discussed in a recent review in detail (13):

a) coagulation by inorganic electrolytes
b) flocculation by polymers
c) carrier flotation
d) agglomeration with the help of hydrophobic substances

Coagulation by Inorganic Electrolytes

Selective coagulation has been used, to the author's knowledge, only on a laboratory scale for the separation of minerals. For example, Pugh and Kitchener (14-16) have obtained some selective coagulation in rutile/quartz and hematite/quartz systems by adjusting the pH

and ionic strength so that only hematite or rutile coagulated. Similar coagulation has also been reportedly obtained for colored impurities from kaolin (17). It would appear worthwhile to study the utility of this technique at least for those ores that do not have too complex a mineralogy.

Selective Flocculation

Selective flocculation of minerals using polymers followed by any one of the separation techniques such as flotation, elutriation, filtration, etc. offers promise for beneficiating mineral slimes. Natural and synthetic high molecular weight polymers have in fact been successfully used as flocculants for clarification of effluent waters in a number of industries. The flocculation processes in mineral processing systems are complicated due to effects and interactions of number of variables as well as the heterogeneity of the ores. Selective flocculation has been successfully used during the last two years on a commercial scale for the beneficiation of low grade iron ore by the Cleveland-Cliffs Iron Ore Company. This process uses reagents which flocculate the hematite leaving the quartz and silicates dispersed. The commercial use of this technology is, however, currently limited to beneficiation of the Tilden iron ore and to that of potash in a Cominco plant in Saskatchewan, where clay flotation is achieved by the joint use of a synthetic flocculant and a cationic collector. Even though the process appears to hold potential for other ores, various problems existing both at the basic and applied level will have to be solved before this potential can be fully realized. For example, a major problem is that most of the currently available long chain polymers are bulk flocculants and lack the desired specificity. As indicated earlier, specificity can, however, be introduced by incorporating active groups into the polymers. Past work on selective flocculation using such modified polymers deals mostly with binary mineral systems in which the valuable mineral was a metal sulfide (galena, pyrite or sphalerite), or a metal or its oxide (hematite, chromite, iron and titanium) and the other component was a gangue mineral. Kitchener *et al*. (8, 18) have made some interesting studies in this regard. They have claimed selective flocculation of sulfide minerals with mercaptan substituted polyols (xanthates). However, use of high molecular weight polymers with substituted mercaptan groups or other specific metal chelating groups as selective flocculants appears to be limited. One such use is that of Clauss *et al*. (19) who reportedly obtained selective flocculation of cassiterite from quartz-cassiterite mixtures under certain conditions using hydroxamic acid substituted polyacrylamide. Mechanisms involved in such process are, however, largely unestablished.

Reports of separation by selective flocculation of multi-component natural ores themselves are scant. In addition to the two commercial applications mentioned earlier, one noteworthy attempt is that of Carta *et al*. (20) for the beneficiation of ultrafine fluorite from

latium. For most systems, however, selective flocculation is not easily achieved even under conditions when excellent selectivity is expected. This fact becomes easily evident upon examining, for example, the results obtained by Usoni et al. (21) during their investigation of the selective properties of anionic, cationic and nonionic polymers as flocculants for several minerals individually and then in combination with each other. They observed the prediction of selective flocculation on the basis of the results from single mineral tests to agree fairly well with the results obtained for pyrite-quartz and sphalerite-quartz mixtures using nonionic Separan but to fail for mixtures of galena-quartz and sphalerite-quartz using anionic Aerofloc or Hercules CMC and for the mixture of Smithsonite-quartz even with Separan which had worked for all other mixtures. Nature of such interactions between minerals is an important problem that deserves a detailed study.

In this regard, an analysis of the role of various reagents and solution conditions that has been selected by U. S. Bureau of Mines for the selective flocculation of taconite ore should help towards developing similar schemes for the beneficiation of other problem ores that are characterized by fine dispersion of values in the matrix. Such an analysis was conducted recently for a discussion of this process at the Engineering Foundation-AIChE Conference on Physical Separations (22). Several questions arose during such an analysis. How exactly do the dispersants work? Why is a particular dispersant, or, as in the present case, a mixture of dispersants, chosen? What is the interaction between the dispersant and the flocculation reagent that is subsequently added? What is the basis for the pH selection? It would be interesting to see whether the conditions chosen for the selective flocculation are in accord with the present theoretical concepts of flocculation and dispersion. On the basis of the criteria discussed earlier, it is generally considered that if the magnitude of the zeta potential of particles is less than 10 to 20 mV, they can aggregate, provided, of course, that there are no other interfering factors such as steric stabilization by macromolecules adsorbed on the particles. It can, therefore, be concluded that selective coagulation of hematite from its mixture with quartz is possible if the zeta potential of hematite is say, -10 to -15 mV, and that of quartz is -30 to -40 mV. Our results for hematite (23) and those of Iwasaki et al. (24) for goethite suggest that above pH 9.0 zeta potential of hematite will be larger (in magnitude) than -20 mV even at as high an ionic strength as 10^{-2}M. On the basis of the reported pzc values for hematite (pH ~8.0) and quartz (pH below 3.0), it should be possible to achieve selective coagulation by operating at a pH of 9 or so in the absence of a flocculant. So why was it necessary to increase the pH to 10.5 - 11 in this process (25)? It is also interesting to consider why it was necessary to add sodium silicate and sodium tripolyphosphate. Does the latter also act as dispersant or only as a sequestering agent to minimize effect of ions such as calcium that are present? Indeed one clear possibility is that it is the selective adsorption

of starch on hematite particles that is responsible for their selective flocculation and levels of other conditions were so chosen in this process so as to minimize adsorption of starch on quartz particles. Is it then possible that the zeta potential of adsorbed starch layer on hematite (which may not be undesirably high) is responsible for producing selective flocculation? To my knowledge, fully substantiated answers to these questions are non-existent.

Another major point that needs clarification is the lack of flocculation of quartz with starch even when hematite or goethite is disseminated in this quartz. Is it likely that starch adsorbed on iron oxide regions of the quartz particles cannot provide complete coverage that is possibly required for aggregation? Incomplete coverage will lead to exposure of the highly charged quartz regions and thereby possibly electrostatic repulsions between them. On the other hand, is it also likely that the iron oxide was selectively removed from the surface regions during agitation of the pulp? We have observed using Auger spectroscopic studies that excessive segregation of quartz is possible on the surface of natural hematite particles (23). In this case, even though the bulk analysis indicated less than 5% of silica in the sample, surface analysis showed the presence of as much as 50% silica under certain conditions. Very little research has been done on the role of composition and heterogeneity of surface layers which is what should be important for adsorption, flocculation, and flotation. Recent development of such techniques as electron spectroscopy for chemical analysis and secondary ion mass spectrometry does make it possible now to obtain information on the chemical composition that one is working with.

Another problem of major importance on which almost no basic work has been reported, is the effect of addition of reagents such as flocculants on subsequent operations such as flotation, filtration, and possibly even pelletizing. Usoni et al. have in fact shown that polymers can enhance or depress flotation depending on the nature, concentration and duration of contact of the polymers with the mineral. With regard to effects of flocculants on filtration, major setbacks can result if the yield strength of the flocculated product is not maintained at a level that can prevent deterioration of permeability during filtration. Again information on such interactions and mechanisms by which they evolve are at present mostly speculative and only future research will provide us with real answers.

Carrier Flotation

Carrier flotation, known also as ultraflotation and piggyback flotation, uses, as the name indicates, an auxiliary mineral as a carrier for the fine particles to be floated. The carrier mineral particles are coarser than the material to be floated. It is speculated that the fines form a slime coating on the carrier mineral which is then floated and the fines are thus piggy-backed into the froth along with the coarser carrier mineral. Ultraflotation has been used by

Engelhard Minerals and Chemicals for more than a decade on a commercial scale to remove anatase from kaolin. While the anatase does not float by itself, it is cofloated with a coarse auxiliary mineral such as calcite (26). This technique, to our knowledge, has not been successfully used for beneficiating any other ore on a commercial scale. Work with phosphatic slime gave limited success in some cases and practically no beneficiation in some other cases. The reasons for the failure of this process in the case of phosphatic slime or for even its success in the case of clay are not understood. Evidently, basic research on mechanisms of ultraflotation is in dire need. The questions to be answered include the following: What is the role of the carrier and its properties? What is the role of other process variables such as intensity of agitation? Does shear flocculation between the fines and coarse play any governing role (3)? Is ultraflotation actually a flocculation process and if so, between what type of particles?

Spherical Agglomeration

Agglomeration with the help of oil has been used by Puddington et al. (27, 28) for graphite, chalk, zinc sulfide, coal, iron ore and tin ore in aqueous solutions. In this process, fines are tumbled in an aqueous solution containing an immiscible liquid which forms capillary bridges between reagentized particles and causes their aggregation. In a similar process, Warren Spring Laboratory is reported to have obtained good separation for a lead/zinc ore (29) owing to hydrophobic bonding between reagentized slime particles. Here a slime fraction of the ore was dispersed by a combination of sodium silicate, sodium carbonate and a polyacrylate and subsequently conditioned with copper sulfate and then potassium amylxanthate was added. Formation of flocs was attributed to similar reasons that gave Gaudin and Malozemoff (30) selective aggregation of galena slimes in the presence of xanthates. On the same basic principle, recently Rubio and Kitchener (31) have reported selective flocculation of mineral fines using hydrophobic polymers after reagentizing the fines with xanthate. Cost of the oil or other reagents required for the capillary bridging seems to be a limiting factor. A complete understanding of the basic role of various parameters such as intensity and type of agitation and chemical interactions at the oil-solid interface and of the effects and interactions of such variables on grade and recovery, agglomerate strength, porosity and the oil recovery might make this process attractive.

RESEARCH NEEDS

Evidently, the potential of the selective aggregation processes is enormous, but the above discussion clearly shows also the enormous gap in our basic understanding of them. Research needs are many in this area and a few major ones are summarized below.

1. Development of heteroaggregation models that take into account the energy of steric interaction between adsorbed layers.
2. Investigation of reasons for lack of selective coagulation even when the zeta potential of the particles are appropriate for it.
3. Investigation of reasons for the failure of predictions for selective flocculation on the basis of single mineral tests.
4. Detailed study of the effects of dissolved ions and the role played by sequestering agents and dispersants.
5. Study of the effect of mineralogical bulk and surface heterogeneity on the degree of separation obtained.
6. Examination of the role of the configuration of the polymer adsorbed on mineral surfaces and the means by which it can be controlled.
7. Elucidation of mechanisms of polymer adsorption and related kinetics.
8. Study of the effects of flocculants on downstream processes such as flotation, filtration and pelletization.
9. Elucidation of mechanisms of carrier flotation; study of the general role of carrier flotation and type and intensity of agitation as well as shear flocculation.
10. Investigation of ionic and molecular mechanisms at solid/oil interface; examination of the influence of interactions between relevant variables on grade, recovery, agglomerate strength, oil recovery, etc. in spherical agglomeration.
11. Possible use of other field forces such as that of magnetic forces in the development of economical selective aggregation processes.

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