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Foam/Froth Flotation

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FOAM / FROTH FLOTATION
Part II. Removal of Particulate Matter

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ABSTRACT

In this second part of the Review on foam and froth flotation, many other aspects of the flotation process are examined, referring mainly to the separation of particulate matter. The effect of particle size is analyzed, stressing the effect of the finer fractions; the role of bubble size is also examined. Among the flotation techniques investigated are those capable of treating fines, such as electrolytic flotation, dissolved-air flotation and column flotation. Operation and design are also discussed. The subject of mineral processing is finally addressed, since it is the main application of the process.

CONTENTS

1. INTRODUCTION
2. PROCESS PARAMETERS
 - 2.1 Effect of Particle Size
 - 2.2 The Role of Bubble Size
3. FLOTATION SURFACTANTS
4. FLOTATION TECHNIQUES
 - 4.1 Electrolytic Flotation
 - 4.2 Dissolved-Air Flotation
 - 4.3 Other Flotation Techniques
 - 4.4 Bacterial Leaching
5. COLUMN FLOTATION
6. OPERATION AND DESIGN ASPECTS
7. HYDRODYNAMICS AND MODELLING
8. FLOTATION IN MINERAL PROCESSING
9. CONCLUDING REMARKS

10. ACKNOWLEDGEMENTS
11. NOMENCLATURE
12. REFERENCES

1. INTRODUCTION

Many industrial processes, at some stage, generate or utilize fine particles ranging in size from over 100 μm to less than 1 μm . It is often required subsequently to remove them from liquid streams, since they contribute to fouling, coking and erosion; they also cause foaming, entrainment and emulsion stabilization problems. Their release may also cause potential environmental problems¹.

This is usually the case in mineral processing; ultra-fine particulate matter, however, exists in water and wastewater, too, needing removal before it is made potable or is discharged. The availability of an effective separation technology is essential, because of the impact on downstream operations and product quality.

Flotation is one of the processes available for the separation of particulate matter from dispersions; it is extensively used in mineral processing, and to a lesser extent in wastewater treatment².

The present paper is the second part of a review of currently available flotation technology; the first part³ dealt with the recovery of metals by ion flotation from dilute aqueous solutions.

2. PROCESS PARAMETERS

The following phenomena affect either recovery or grade or even both in flotation :

- | | |
|------------------------------|--------------------------------------|
| a) high specific surface | i) coagulation / agglomeration |
| b) high reagent consumption | j) low momentum |
| c) high suspension stability | k) fine particle entrainment |
| d) froth stability | l) low collision probability |
| e) high surface energy | m) slime coating |
| f) small mass | n) low flotation rate |
| g) rapid oxidation | o) low adhesion probability and |
| h) high dissolution rate | p) non-specific collector adsorption |

Most of them are interrelated in many ways.

2.1 Effect of Particle Size

Flotation relies heavily upon the successful collision and adhesion of the particles onto air bubbles; hence the size of the particles must play an important role in the process.

The variation of flotation recovery with particle size in industrial concentrators was first studied back in 1931 by Gaudin and coworkers; they collected flotation data on copper, lead and zinc ores in a series of cells and correlated cumulative recoveries with particle size.

In mineral processing, size reduction is used to liberate valuable material from gangue⁴; this usually produces a substantial amount of fines, since ores may be classified as either those in which the desired mineral occurs almost entirely in the fine size range, or those in which the

TABLE I
Observed size range for maximum flotation recovery ⁸.

Mineral	Size range (μm)	Conditions
baryte	10-30	(LB)*
cassiterite	3-20	(I)*
fluorite	40-110	(LB)
	10-90	(I)
	50-150	(I)
galena	37-295	(LB)
	170-240	(LB)
	7-70	(I)
	6-70	(I)
	13-75	(I)
	20-100	(I)
pyrite	50-150	(LC)*
pyrite-pyrrhotite	20-70	(LB)
quartz	10-40	(LC)
	9-50	(LB)
sphalerite	15-100	(I)
wolframite	20-50	(LB)

* L : laboratory, I : industrial, B : batch, C : continuous

mineral extends from coarser sizes down to the fines. The ultrafines problem has been reviewed by Somasundaran ⁵, while the recovery of very fine particles in mineral processing was recently discussed ^{6,7}.

Fuerstenau, while reviewing the floatability of fines ⁸, reported the observed size ranges of maximum flotation recovery, which are given in Table I. Maximum recovery was noticed in the range 10-100 μm ; he also suggested that chemisorption improved fines recovery for the following reasons :

- specific chemical interactions maintain selectivity,
- the free energy of adsorption is large, resulting in lower reagent consumption;
- the electrical charge on the mineral is lower - resulting in better recovery - if collector adsorption occurs by exchange or neutral molecule adsorption; and
- the reduced adsorption of collector on bubble surfaces contributes to better recovery of fines.

Many postulates have been proposed to describe the behavior of fines in flotation ⁹, as for example : diffusiophoretic motion, attachment through rotation of angular particles, increasing dynamic contact angle, surface age, increased solubility, increased surface stress, increased rate of adsorption, low particle momentum etc. Experiments with two different-sized pyrite fines were conducted in a Hallimond tube with dispersed air ¹⁰. Figure 1 illustrates the fact that their behavior, as related to the pulp pH, is not quite similar. In this case, sodium hydroxide (and sulfuric acid) solutions were used for pH modification. Hydroxides are well known as pyrite depressants ¹¹, depending on the collector applied.

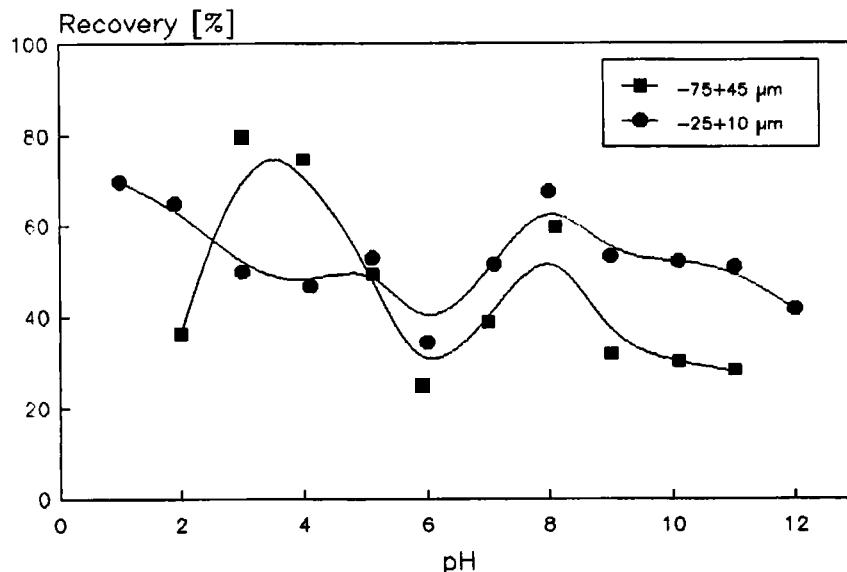


FIGURE 1

Influence of pH on pyrite recovery for two size ranges (flotation in a Hallimond tube). [Potassium ethyl xanthate] = 15 ppm; pulp density : for the coarser size, 1%; for the finer size 5% Reprinted from ref. (10).

Foam flotation was also applied to the separation of NaY zeolites from an aqueous suspension¹²; the zeolites had been used previously for the removal of zinc ions from the aqueous solution. The particle size range of the zeolites was in the range +1.9-5.5 μ m; Figure 2 shows that flotation is capable of operating even for such fine particles (a concentration of 1×10^{-5} M of either surfactant was found to be satisfactory for the flotation of the synthetic zeolite).

Many theoretical papers examine the relation of fine particle flotation and kinetics¹³⁻¹⁶. In general, the flotation rate constant (k) has been found to depend upon the bubble and particle size according to

$$k \sim \frac{d_p^m}{d_b^n}, \quad (1 < m < 2 \text{ and } 2.5 < n < 3)$$

where d_p and d_b are the particle and bubble diameters, respectively.

Serious consideration should be given to devising separate flotation circuits designed specifically to handle the fine size ranges. The cells could be agitated at the minimum speed for complete suspension of all particles to obtain the optimum recovery. It has been suggested that specific flotation methods should be applied to the processing of fine particles¹⁶⁻¹⁸.

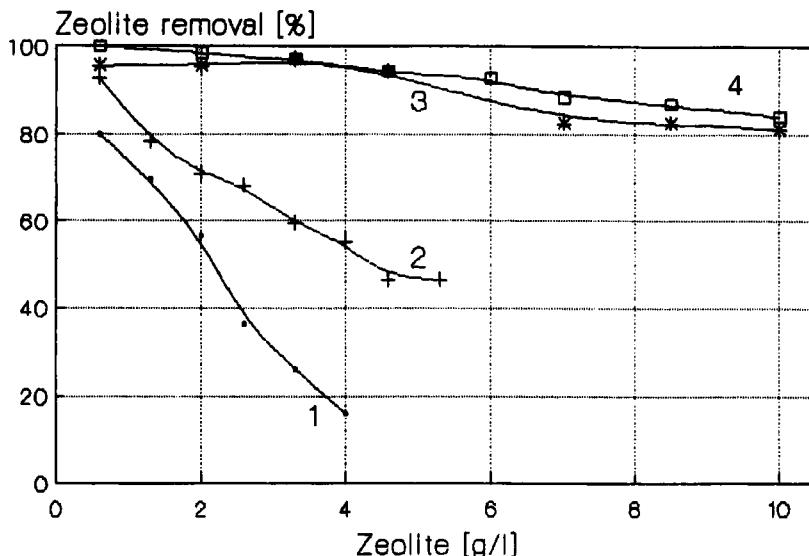


FIGURE 2

Effect of collector concentration and initial zeolite concentration on foam flotation. (1) 1×10^{-5} M cetyl trimethylammonium bromide; (2) 0.4×10^{-5} M laurylamine; (3) 5×10^{-5} M cetyl trimethylammonium bromide; (4) 2×10^{-5} M laurylamine. Reprinted with permission from ref. (12); copyright Marcel Dekker.

2.2 The Role of Bubble Size

In practice, it is difficult to reduce bubble size without affecting other variables in the system. It is for this reason, probably, that few studies have been published concerning the effect of bubble size on the industrial-scale flotation of fine ore pulps.

Although fine bubbles will improve certain aspects of the process, no general conclusion may be drawn as to whether or not the use of fine bubbles will improve the overall results of any flotation system; each one must be studied individually.

It has also been suggested that at constant bubble size, the collision rate decreases with decreasing particle size¹⁹; if the bubble size is reduced in proportion to the particle size, the rate of collision remains unchanged. Eigeles²⁰ proposed that there is an optimum relationship between the diameter of the air bubble and the particle.

Actually, according to our knowledge, Volkova was the first one to study, back in 1940, the role of bubble size²⁰, while investigating the floatability of calcite particles. Using photographs, she showed that bubbles were mineralized preferentially by small 45 μm particles rather than larger ones (100-150 μm); larger grains (150-200 μm) were floated by a group of bubbles. Further addition of oleic acid (collector) permitted the flotation of particles

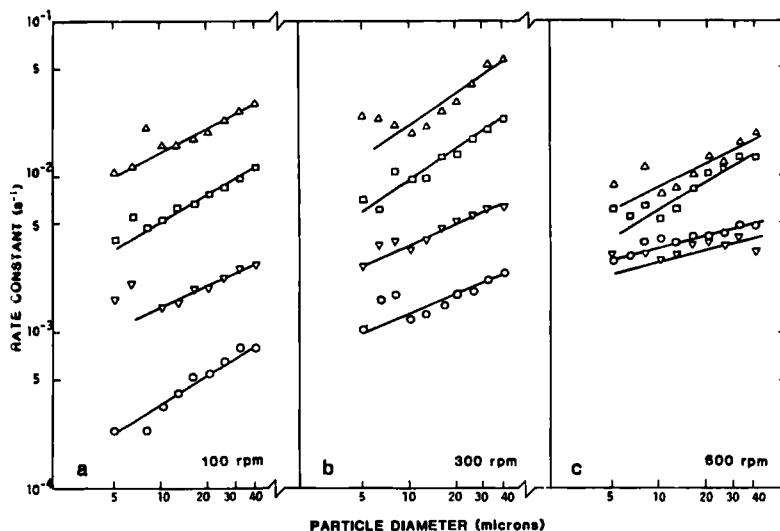


FIGURE 3

Rate constants for quartz plotted as a function of particle diameter, stirrer speed and bubble diameter. Bubble size: Δ , 75 μm ; \circ , 165 μm ; ∇ , 360 μm ; \square , 655 μm . Reprinted with permission from ref. (17).

by much smaller bubbles. She concluded that the attachment of particles to bubbles depends upon the relative dimensions of particles and bubbles.

In another study, the rate constant for quartz flotation was plotted against particle diameter for various stirring speeds¹⁷ (Figure 3); the rate of flotation of fines was accelerated considerably by using very small bubbles of less than 100 μm . It should be noted that conventional froth flotation cells usually produce air bubbles in the size range of 0.6-1.0 mm.

Two methods, dissolved-air and/or electrolytic flotation, which will be briefly reviewed below, are capable of producing fine bubbles; Figure 4 illustrates the size of bubbles obtained during electrolytic flotation²¹. It was noticed, unexpectedly, that the statistical mean and the standard deviation on the empirical distribution, with respect to the bubble size, decreased as the density of the current applied increased. In the meantime, it was found that the number of bubbles was also increasing.

Recently, it was suggested that many of the problems encountered in separating fine particles could be alleviated by using significantly smaller air bubbles than those used in conventional flotation processes²². The main advantage of using smaller bubbles may be found in an increased recovery rate and possibly selectivity, which may be attributed to an improved bubble-particle collision efficiency.

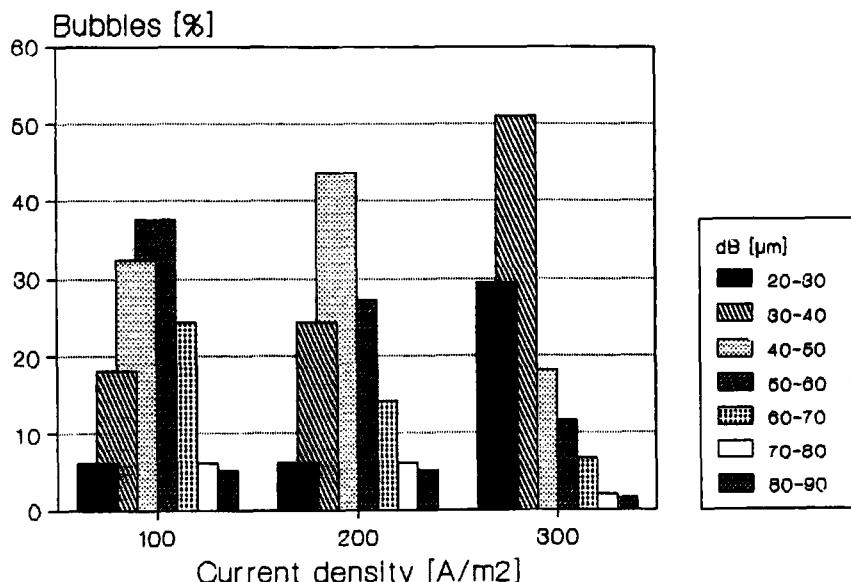


FIGURE 4

Distribution of electrolytically-produced gas bubble size in flotation conditions observed at various current densities applied. Reprinted from ref. (21).

3. FLOTATION SURFACTANTS

These involve both inorganic and organic reagents; they can be distinguished in *collectors*, the most widely used class, *frothers*, and *modifiers*. According to the electrical charge associated with their polar group, they are also classified as *anionic*, *cationic*, or *nonionic*; in the latter belong the frothers, flocculating agents, depressants etc. Finally, according to another classification, they are distinguished in *thio* (or *sulphydryl*) and non-thio, ionizable components. This classification is due to the fact that the former act primarily as collectors for metallic sulfides, the dominant field of flotation in mineral technology. Examples of common collectors are given in Table II - see ref. (23) for more details.

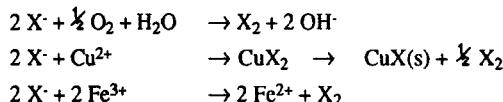
The only variety of cationic collectors used in industry is the amine type (not included in Table II), classified as primary, secondary, tertiary amines or quaternary ammonium salts. Their application to ion flotation has already been discussed in Part I³, together with fatty acids (derivatives of carboxylic acid). The latter constitute a common group of non-thio surfactants of anionic type.

An important characteristic of surfactants is their solubility³. Another interesting point is the oxidation of collectors, particularly the thio-type ones. In addition to oxidation by cupric and

TABLE II
 Common flotation collectors and their chemical formulae
 (R represents the hydrocarbon chain - alkyl, aryl or cyclic).

Alkyl carboxylate (as Na or K salts)	
Alkyl sulfonate	
Alkyl sulfate	
Alkyl hydroxamate	
Mercaptans	RSH
O-alkyl-monothiocarbonate	
Alkyl xanthate	
Alkyl trithiocarbonate	
Dialkyl dithiocarbamate	
Alkyl thionocarbamate ester (where R ₁ = C ₃ H ₇ , R ₂ = C ₂ H ₅)	
Dialkyl dithiophosphate (aerofloats)	
Mercaptobenzothiazole	

ferric ions, xanthates (i.e. O-alkyl-dithiocarbonates) can also be oxidised to the dimer, dixanthogen, by dissolved oxygen, following the reaction²⁴



Dixanthogen is necessary, for instance, for the flotation of pyrite²⁵.

New collector types are also being tested, such as modified thionocarbamates, dithiophosphinates, organic sulfides, trithiocarbonates, monothiophosphates and modified thioureas.

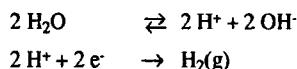
The adsorption of surfactants from aqueous solutions is a phenomenon of central importance in applications ranging from ore flotation to water treatment. It is a process of such considerable complexity that there is a tendency in the literature to present a more or less new model for surfactant adsorption every time a new application is considered.

The mechanisms of collector adsorption-abstraction (for ionic and non-ionic surfactants) on heterogeneous surfaces was recently reviewed²⁶.

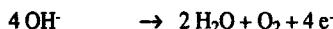
4. FLOTATION TECHNIQUES

4.1 Electrolytic Flotation

This bubble generation method is based upon the electrolysis of water; cathode reactions :

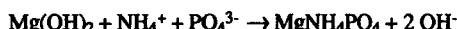


and anode reaction



Chlorine may also be produced, depending upon the water salinity.

One of the first application of the method was for sewage mixed with seawater in Oslo²⁷, with a cell separated by a diaphragm. Only the cathode section was used, at the time, with the additional advantage that the following useful reaction was also taking place :



Various types of flotation apparatus were tested in the (then) USSR such as electrolytic preparation of slurry with simultaneous flotation, or electrolytic preparation in a vat followed by flotation in a separate cell²⁸.

In fact, the electrodes chosen should satisfy several criteria²⁹ : no electrode corrosion, avoidance of scaling, operation at high current densities, and no production of objectionable gases.

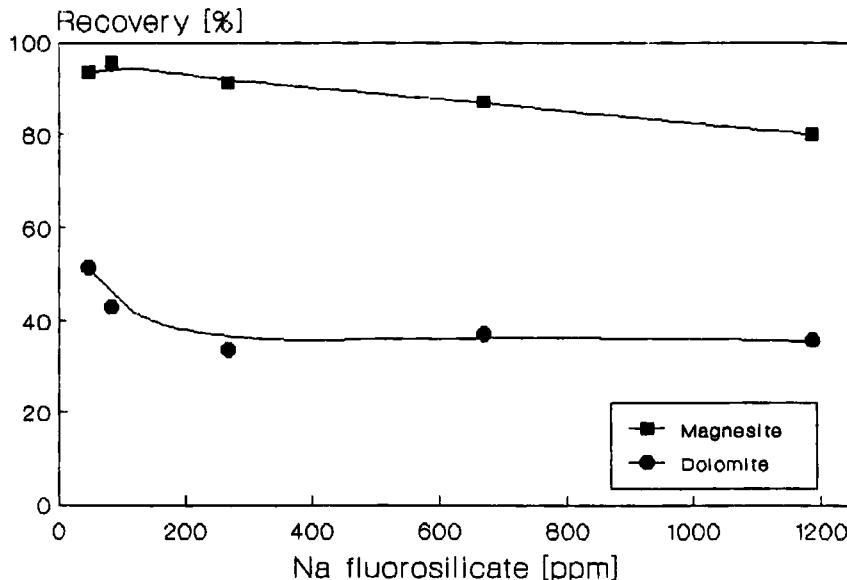


FIGURE 5

Effect of the amount of sodium fluorosilicate added as modifier on the electrolytic flotation recovery of carbonate fines. pH 11.6, [collector] (tall oil fatty acid) = 40 ppm. Reprinted from ref. (34).

Suitable electrodes for the process have been described¹⁸:

- i) Si-Fe (16%-84%), which has a life of 28 days in a 0.1% NaCl solution;
- ii) platinised titanium and, mainly, titanium covered by lead dioxide; these operated for more than two years in severe conditions; and
- iii) Pt - 10% Ir alloy wire spot-welded to a Columbium substrate, which was found to be stable under 90V in seawater, with an anode corrosion rate of 2.54 μm per year.

Various electrolytic cell designs were constructed and tested³⁰, including horizontal perforated sheets made of stainless steel or mild steel and graphite, grid-expanded mesh, bipolar cells with vertical plates, and a membrane-separated electrode cell.

The technique of electrolytic flotation has been applied for various mineral systems, such as pyrite fines (using amyl potassium xanthate as collector³¹; recovery was better when only electrolytic oxygen was present), manganese slimes, cassiterite fines³², clay minerals (using sodium oleate), enargite fines³³ and fine diamond tailings. Figure 5 presents some of the results obtained when treating magnesium carbonate fines (magnesite, MgCO_3 and dolomite, $\text{Mg,Ca}(\text{CO}_3)_2$; size range -45+15 μm), using sodium fluorosilicate as a modifier³⁴; a remarkable depressing action on the calcium mineral was noticed and this could possibly be used for selectively separating the two minerals.

Sodium silicate was found in classical flotation to depress dolomite (at a pH < 9.0)³⁵; it had also a slightly activating effect on magnesite. When sodium silicate was used as a modifier

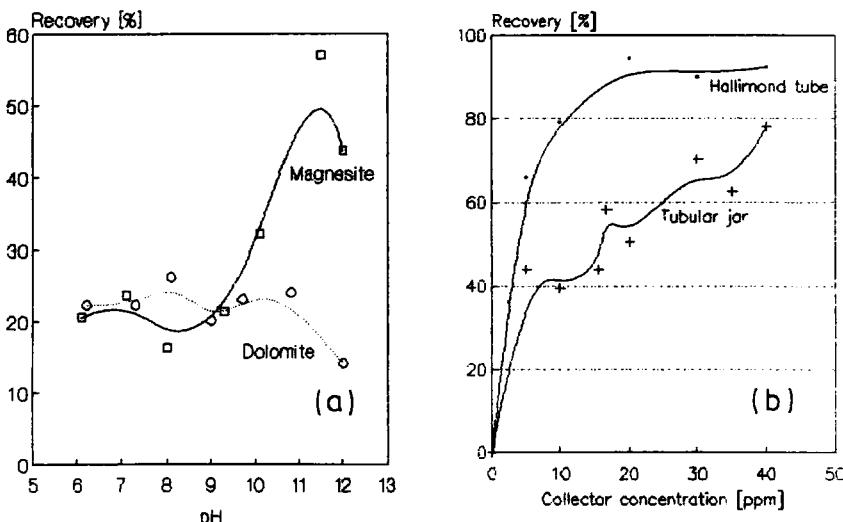


FIGURE 6

(a) Effect of the addition of sodium silicate (100 ppm) as modifier on the collectorless electrolytic flotation of fine minerals. Reprinted from ref. (34). (b) Effect of collector (fatty acid) concentration on the (electrolytic) flotation of dolomite (A : cylindrical jar; B : Hallimond tube). Reprinted from ref. (36).

(Figure 6), a difference of approximately 40% in recoveries of the two minerals was noticed, at a pH value of approximately 11.5; dolomite was again depressed. The most important feature of these experiments lies in the total absence of collector, which is an advantage of electrolytic flotation. In the same figure, the effect of collector addition (Acintol FA-1 tall oil from Arizona Chemical Co.) on dolomite flotation is also illustrated for two different electrolytic flotation cells ³⁶. The various applications of electrolytic flotation to minerals are summarized in Table III.

The effects of hydrogen and hydroxyl ions on flotation, concerning mainly their effect on the surface hydration in the absence of collectors, the surface coverage by the collector, and the adsorption of collector was discussed elsewhere ²⁰. However, phenomena needing further examination include : the adsorption of H^+ and OH^- on the mineral, the change in the ionic composition of the pulp, the displacement of collector from the surface, the change in the active form of collector, competition of H^+ and OH^- with flotation-active collector ions and the change in the electrical properties of the surface. The application of electrolytic flotation in recovering metal ions from solutions was covered in the first part of the review ³.

A final word about electrolytic flotation : when compared to the conventional process, it brings about additional expenses, mainly for the energy required by the electrolysis. However,

TABLE III
Applications of Electrolytic Flotation ³².

baryte	manganite
cassiterite	molybdenite
chalcopyrite	montmorillonite
chromite	phosphorite
clays	psilomelane
copper sulfides	pyrite
diamond	pyrolusite
galena	quartz
gold	sphalerite
limonite	sulfides

these expenditures are compensated in most cases by :

- lower consumption or even complete elimination of collector;
- improved flotation rate;
- better concentrate quality;
- possibility of complete utilization of raw materials; and
- utilization of raw materials that cannot be beneficiated by any other existing process.

4.2 Dissolved-Air Flotation

The method finds many applications in effluent treatment ². The wastewater usually flows from a flocculation tank under quiescent conditions into the centre of a 3 m deep circular flotation unit, where it is discharged, about 1.5 m from its base, through a pipe with equi-spaced holes ³⁷. Pressurized recycle water is introduced into the unit, approximately 0.5 m below the wastewater feed, through a circular pipe fitted with nozzles.

The flotation activation of mineral particles by minute bubbles is well known ²⁰, mainly for the following reasons :

- a) a small bubble acts as a spearhead on the surface of particles, aiding attachment;
- b) the stability of the hydrated film separating the colliding large and small bubbles is much lower than that between the bubble and mineral; and
- c) the large difference in the capillary pressures inside the colliding bubbles is an additional force utilized in the disruption of the film.

The influence of dissolved gases was also discussed ²⁰, mainly the precipitation of gases from solution, the change of pH of the medium, the adsorption of gases on the mineral grain surfaces, as well as their surface oxidation. Other phenomena affected by dissolved gases are the hydration or dehydration of the surface (in the absence of collector), the change in adsorbed reagents and the change in ionic content of the pulp.

Figure 7 illustrates the major mechanisms responsible for dissolved-air flotation, mainly :

- a) the adhesion of gas bubbles on the particle surface,
- b) trapping of bubbles within a floc structure during the rise of the bubble,
- c) adsorption of bubbles in a floc structure as the floc is formed, and
- d) formation of bubbles within flocs.

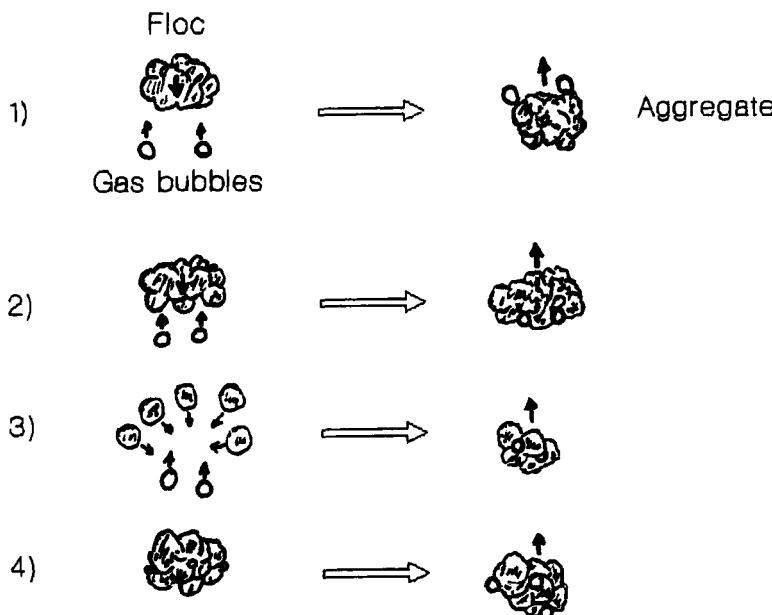


FIGURE 7
Illustration of major mechanisms responsible for dissolved-air flotation.

Certainly, the third mechanism does not take place in dispersed-air flotation, where the bubbles are much larger.

The study of electrokinetic properties of salt-type minerals is also important for flotation, since the adsorption of the various inorganic or organic ions from the pulp is governed from the place of the point of zero charge (pzc), in regard with the concentration of ions which determine the potential in the system under study. Figure 8 illustrates the results obtained while processing magnesium carbonate fines by dissolved-air flotation, using a commercial fatty acid collector and tetrasodium pyro-phosphate (TSPP) as a modifier³⁸. Measurements of ζ -potential taken in parallel are shown in Figure 9. Dolomite showed greater negative values than magnesite, although no sign reversal was observed; a pzc around pH 6.5 (under the same conditions) was found.

The fatty acids removal from effluents by mineral fines (magnesite and dolomite) was also investigated with dissolved-air flotation³⁹, using again TSPP as a modifier, but at pH 11.0; since it is known that phosphates depress dolomite flotation in the alkaline region. This may be attributed to adsorption on sites where calcium exists. It should also be noted that, although the two minerals were in the same nominal size range, their respective specific areas were very different.

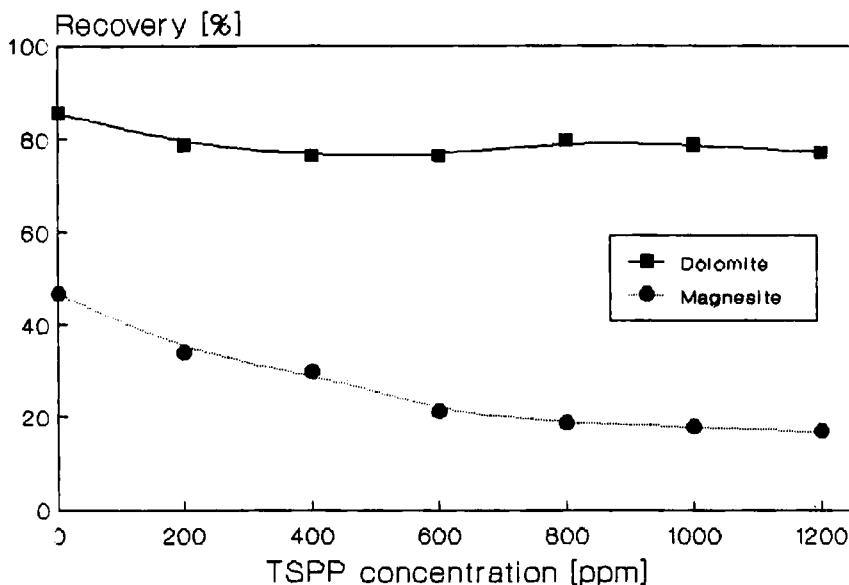
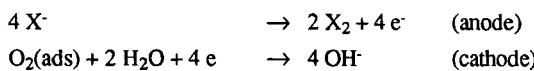


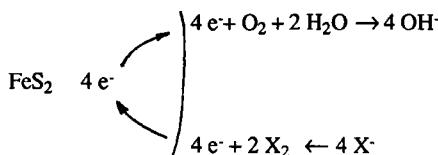
FIGURE 8

Effect of tetrasodium pyrophosphate (TSPP) addition on the dissolved-air flotation of fine minerals. pH 7.1, [collector] (tall oil fatty acid) = 40 ppm. Reprinted with permission from ref. (38); copyright Elsevier.

Pyrite fines (-25+10 μm) were treated both by dissolved-air and dispersed-air flotation, using potassium O-ethyl-dithio-carbonate (xanthate) as a collector^{10,40}; a similar behavior was noticed in both cases. From the various xanthate species, it is known that dixanthogen is responsible for the flotation of pyrite; it is formed by the following reactions:



where X is the xanthate ion. Since sulfide minerals conduct electricity, the electrons are transferred through the solid phase:



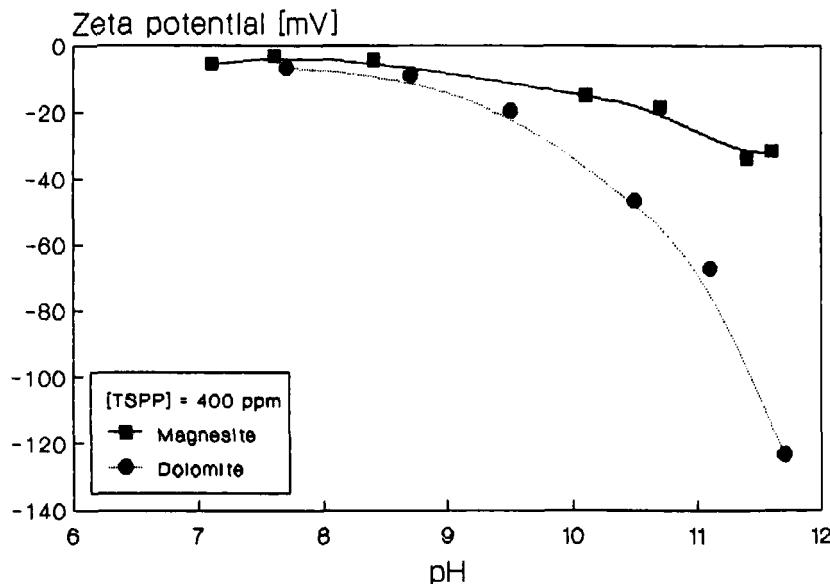


FIGURE 9

Zeta-potential measurements (by the mass transfer principle) of fine particles in the presence of tetrasodium pyrophosphate (TSPP). Reprinted with permission from ref. (38); copyright Elsevier.

4.3 Other Flotation Techniques

Several other minor flotation techniques could also be described :

- the use of an oil phase; depending upon the dosage of oil, different processes may be distinguished, from simple flotation with oil as a collector, extended to agglomeration (emulsion) flotation. Low-grade iron ore has been successfully beneficiated.
- carrier flotation (or ultraflotation). It has been postulated that the fines form a coating on the carrier mineral and are then floated with the coarser carrier particles. Oleate flotation of ultrafine anatase (TiO_2) impurity from clay using an auxiliary material, such as calcite ($CaCO_3$) is an example.
- preaggregation of the fine particles before the actual flotation process, if the decrease in the efficiency of separation is attributed to such factors as lower collision and adhesion rates of fines.

Conventional froth flotation may also be modified and extended to lower size ranges possibly by other means, like :

- the adjustment of some process variables, like e.g. temperature : at Cleveland Cliffs Iron Company, the conditioning of iron ore fines pulp at elevated temperatures improved the plant performance ⁸.

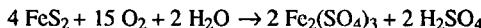
the use of specifically-adsorbing flotation agents.

Several other flotation techniques could also be included under this classification, e.g. precipitate flotation or adsorbing colloid flotation, solvent sublation and others.

Although in practice the existence of a lower particle size limit is well known, however it does not appear that we are near intrinsic limits, since even ions may be selectively floated, under different conditions.

4.4 Bacterial Leaching

The technology of bioextraction has been known for some time, having been used successfully for the extraction of copper from waste dumps and uranium from worked-out underground mines. The treating of ores by processes depending on the action of *Thiobacillus ferrooxidans* and other bacteria is now a well established practice. For instance, the bacterial oxidation of pyrite has been described⁴¹ by the following reaction



Today, attention is paid to biochemically-enhanced flotation^{42,42a}. The continuous-flow bacterial leaching of chalcopyrite was reported⁴³, together with economic aspects of the process; chalcopyrite in leach residues was recovered by flotation, reground and re-leached, in order to obtain an overall extraction of over 96%. In this example, biohydrometallurgy and flotation were applied in series, aiding each other.

Bacterial leaching has also been compared to bacterial conditioning and flotation, while studying the desulfurization of coal⁴⁴.

5. COLUMN FLOTATION

Flotation columns are probably the latest development in flotation cell technology, although they were really developed back in the '60s⁴⁵. They were proposed for the treatment of fine particle systems, as an alternative to agitated cells, since the latter perform badly when part of the particulate system treated consists of fines.

The mineral industry was reluctant to adopt this new technology in the beginning, because it thought that it was easier to deslime the ore prior to flotation. However, since this resulted in significant losses of valuable material and, at the same time, it became obvious that increasingly "difficult" ores had to be treated, they started being popular.

They are based on the principles of bubble columns, which are widely used in chemical engineering practice as chemical reactors. Their advantages over agitated cells lie mainly on two factors :

- the longer retention time of the particles, which increases the probability of collision and adhesion of the fine particles on the bubbles, and
- the addition of "wash" water on top of the column, which results in an increased-grade concentrate, since by carefully adjusting the wash water flowrate, a net downward water flow is obtained (termed "positive bias") and most of the entrained gangue particles are washed away from the froth and the concentrate.

TABLE IV
Uses of Flotation Columns.

Minerals treated	Dimensions	Ref.
Zn, Pb, Mo, Cu, Cu/Ni, Au/Fe, graphite	var.	53
Cu/Zn/Pb	14cm x 10m 20cm x 10m	54 54
Au	2.5m x 12m 3.25m x 10m 2.5m x 12m	55 55 50
Coal	10.2cm x 4.3m 10cm x 8.2m	50 56
Cu	10cm x 8.2m	56
Mo	10cm x 8.2m	56
P ₂ O ₅	5.2m x 6.0m	56
Pb	2.5m x 13m	55
U ₃ O ₈	5.2m x 6.0m	56
Zn	2.5m x 13m 1.8m x 12m	55 54

One of the early applications of column flotation was the cleaning of coal ⁴⁶. This has both economical and environmental benefits : since as much run-of-mine coal is recovered as possible, a reduction of air and water pollution is obtained.

Laboratory-scale studies of columns include : studies of the column hydrodynamics ^{47,48}, the use of calcite fines in order to study the fundamental phenomena occurring in the column ⁴⁹; pilot-plant and full-scale operation of flotation columns for various mineral systems has also been reported ⁵⁰⁻⁵². Table IV presents the various mineral systems investigated.

The use of flotation columns is becoming now increasingly popular : many plants are replacing agitated cell circuits by columns, achieving similar or even better grades and recoveries, while at the same time getting energy as well as operating-cost savings ⁵⁷.

Lately, a new type of column has been designed : the Jameson cell ^{50,58}. This consists of a short tubular vessel and a 3m-tall downcomer, through which pulp and air are pushed together. The vessel, in fact, consist only of a froth zone; its performance is claimed to be much higher than either mechanical cells or conventional flotation columns (Figure 10) and other advantages exist, too : the size of the vessel permits an easy operation, and due to the downcomer configuration, the air blower may be discarded, since air is aspirated into it.

6. OPERATION AND DESIGN ASPECTS

Initial floatability tests of a particulate matter are usually carried out in a bench-scale micro-cell of the modified Hallimond tube type ^{35,59}. This apparatus offers certain advantages,

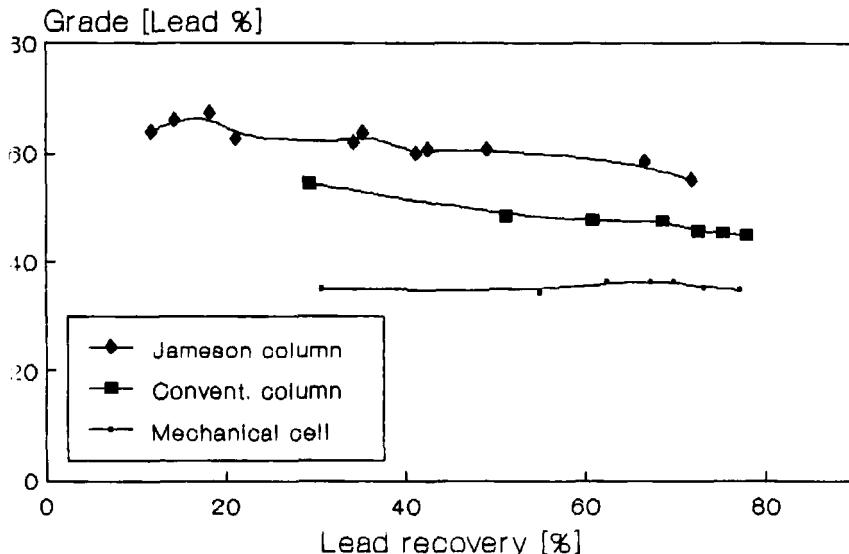


FIGURE 10

Comparison of the metallurgical performance of the Jameson column to mechanically-agitated cells and conventional flotation columns. Redrawn with permission from ref. (53).

particularly is a pure mineral is under study; however it has been said that "... a 1 kg dry charge had been found by experience to be the minimum necessary to keep sampling errors within acceptable limits ..." ⁶⁰.

Current laboratory froth flotation cells are made by : Denver, Wemco-Fagergren, Agitair, Wedag, Outokumpu, Minemet, Mekhanobr etc. Scale-up from laboratory-scale up to the largest commercial sizes (of over 70 m³) evolved on an empirical trial-and-error basis rather than based on first principles and scientific knowledge, mainly due to the importance of the process in the minerals industry.

Harris considered four main topics in flotation machines scale-up ⁶¹ :

- the geometry of the tank and the impeller/stator assembly;
- the speed of the impeller;
- air flow rate and air residence time;
- power consumption and power intensity.

In sub-aeration cells, the impeller is required to perform the triple function of pulp circulation, particle suspension and aeration. The addition of a frother is often necessary to control the bubble size.

Mechanically-agitated flotation cells are often partially or fully-baffled mixers; they are usually analysed in terms of their hydrodynamic characteristics with a series of dimensionless numbers, such as the power number, Reynolds, Froude, Weber, air flow and discharge flow

numbers⁶², each one having a physical meaning⁶³. These conventional numbers, coming from mixing in the chemical processes, are the following :

$$\text{Power number} \quad N_p = \frac{P}{\rho N^3 D^5} \quad (1)$$

$$\text{Reynolds number} \quad N_{Re} = \frac{\rho N D^2}{\mu} \quad (2)$$

$$\text{Froude number} \quad N_{Fr} = \frac{N^2 D}{g} \quad (3)$$

$$\text{Weber number} \quad N_{We} = \frac{\rho N^2 D^3}{\sigma} \quad (4)$$

$$\text{Air flow number} \quad N_{QA} = \frac{Q_A}{N D^3} \quad (5)$$

$$\text{Discharge flow number} \quad N_{QL} = \frac{Q_L}{N D^3} \quad (6)$$

where N is the rotating impeller speed, P the power consumed, D the impeller diameter, ρ the liquid specific density, μ its viscosity, g the gravitational acceleration, σ the gas-liquid surface tension, Q_A the air volumetric flowrate and Q_L the (volumetric) flowrate of the flow discharged by the impeller. The impeller peripheral (tip) speed is also an important parameter.

Basic geometrical features are : the cell height (H), width (W), length (L), impeller diameter and impeller clearance from the bottom of the cell. From these, useful parameters may be computed, e.g. the froth area ($A = W L$), the impeller aspect ratio ($R = D/L$) etc. Geometrical similarity of different-size cells leads to relationships of the following type⁶⁴

$$V \sim L^n$$

$$A \sim L^2$$

$$R \sim D^r$$

where $2 < n < 3$ and $-0.25 < r < -0.5$.

The introduction of a stream of air, to be dispersed in bubbles that will be responsible for flotation, complicates the matter. The use of frothers, surfactants at the air/water interface makes flotation rather peculiar even in the study of gas dispersions. The main points outlined by the late Barbery⁶⁴ concerned, among others : the effect of agitator characteristic on gas hold-up, power consumption in aerated pulps, flooding of the reactor by high air flowrates and the bubble size distribution resulting from the dispersion of the air stream.

Flotation is certainly a three-phase system, which is quite specific in its mixing requirements, as far as particle size and air dispersion is concerned. Similar types of operations are encountered in leaching in the presence of dissolved gases and fermentation. Nevertheless, in these systems the mixing requirements are totally different and more related to optimizing mass transfer for chemical reactions.

Most of the data available today have been gathered and correlated mostly by equipment manufacturers⁶⁰. Average design and performance parameters for commercial scale machines and trends as machine size increases are as follows :

- cell configuration : between constant geometry and constant depth
- speed : $5.6 - 9.6 \text{ m s}^{-1}$ (increases slightly)
- aeration : $10.1 \times 10^{-3} - 30.5 \times 10^{-3} \text{ m}^3 \text{ s}^{-1}$ per m^2 of froth surface;
- power intensity : $52.6 - 210.6 \text{ kW m}^{-3}$ (consumed; decrease slightly);
- capacity : $28.2 - 70.6 \text{ t d}^{-1} \text{ m}^{-3}$.

Typical values for dimensionless numbers are

- | | |
|-------------------|---------------|
| - power number | 0.5 - 5 |
| - Reynolds number | $10^5 - 10^6$ |
| - Froude number | 0.1 - 5 |
| - air flow number | 0.01 - 0.1 |

The basic functions of a mechanically-agitated froth flotation cell are the following :

- a) to maintain particles in suspension,
- b) to disperse air flow into bubbles in the slurry, and
- c) to create the conditions required for contact between air bubbles and hydrophobic particles.

Moreover, the design of the cells must be able to fulfil certain criteria⁶⁵ :

- a) transferring of particles from the slurry feed to the discharge points (either the froth for the concentrate or the next cell for the hydrophilic particles, as well as for the hydrophobic ones that did not adhere to a bubble) should be done relatively rapidly, otherwise they may be ground to finer sizes by attrition occurring in the impeller region;
- b) a region of decreased turbulence should be allowed for at the top of the cell, so that the froth layer is not disturbed, or the particles may detach from the bubbles and re-enter the pulp; and
- c) the design of the cell should be such as to avoid sedimentation and impeller sanding : these are the main causes of motor destruction on starting-up of the cell.

The turbulent fluctuations of velocity prevailing in flotation cells have a strong influence on the results obtained. By correctly selecting the rotor speed in a circuit, optimum results may be obtained.

From the energy consumption point of view, the most economical cell shape is, according to Falleni⁶⁶, the deep cell. Using published data and calculating the parameter ND^2 , he drew P/LW curves for various cell types, as well as graphs of power consumption vs. unit cross-sectional area. The differences in these curves depend probably on the efficiency of the mechanism and the cell in generating eddies in the cell's turbulent spaces, which constitute predominantly the cell space where flotation takes place. Manufacturers solve the various problems in particular ways, which are usually explained in their brochures.

Agitatorless cells exist as well, used particularly in effluent treatment, as for instance : the dissolved-air flotation units, the Davra cell⁶⁷, both belonging to the pneumatic type of cells etc.

The use of mixing during dissolved-air flotation (in effluent treatment) is rather limited. Stirring has been used as a means of reducing the wall effects⁶⁸; this may be beneficial, but on the other hand it was found to be detrimental because it strips air from the sludge floc. The latter may depend on the characteristics of the sludge and the tenacity with which the air bubbles are held to floc particles.

Conventionally, froth flotation machines are grouped in banks of cells in series, which from the feed point onwards are called rougher and scavenger; the rougher concentrate is reprocessed in cleaner circuits⁶⁴. Lately, there is a tendency to develop large flotation cells⁵⁷, having volumes exceeding 70 m^3 . The drive towards very large flotation machines is a

response to greater throughput requirements due to increased demands but lower grades and to increased plant costs.

7. HYDRODYNAMICS AND MODELLING

A flotation unit is fundamentally a form of reaction vessel and consequently a detailed knowledge of the flow characteristics involved in a particular tank may be expected to constitute an important feature of its design, the efficiency of the process depending largely upon the hydrodynamic characteristics of the system.

A model is usually constructed to describe the process happening in the cell; experimental results from residence time distribution (RTD) measurements are used to calibrate it and the model may then be used to predict the cell performance, avoiding lengthy and perhaps expensive plant trials.

The simplest way of modelling a flotation cell is the one-parameter tank-in-series model⁶⁹. The model assumes that the cell may be represented by a series of fully-mixed tanks (CSTRs) connected in series; fitting the experimentally-obtained RTD to the model prediction allows for the determination of the number of ideal cells that seem to model best the cell.

This was applied first to electrolytic flotation; the experimental results showed that the process was indeed nearer to a single CSTR than to a tubular (plug flow) reactor, although quiescent conditions prevailed⁷⁰.

More complex models may consider the real reactor as consisting of different regions of plug or backmixed flow with regions of deadwater or bypassing; recycling and crossflow may also be allowed. A theoretical mixing model, developed by Levenspiel⁶⁹, has the following form for the age distribution function :

$$I = \frac{Q_1}{Q} \exp \left(- \frac{Q_1}{Q} \frac{V}{V_B} \left(\theta - \frac{V_p Q}{V Q_1} \right) \right) \quad (7)$$

as illustrated in Figure 11, where :

- Q_1 is the flowrate of the dispersion (effluent) passing through the tank,
- Q is the total volumetric flowrate of the feed
- V is the total volume of the flotation cell,
- V_B is the backmixed flow volume,
- V_p is the plug flow volume, and
- θ is the dimensionless time, which is the time divided by the mean residence time, τ , of the effluent in the vessel.

The model was applied to electrolytic flotation in effluent treatment^{70,71}; the output corresponding to a step input of a tracer was measured, the ratio C/C_0 (termed the F-curve) calculated and the internal age distribution of the tank contents was found from : $I = 1 - F$. The I function drops from 1 to 0 and the area under the I-curve is equal to $(V - V_D)/V$, where V_D is the deadwater volume. Figure 12 presents the results. From this continuous flow investigation, it was concluded that the plug flow region was of the order of 12% of the volume. In co-current operation (the inlet is near the bubble generator and the outlet near the top of the tank), deadwater region appeared of the order of approximately 7%; this was not noticed in counter-current operation. In both modes of operation, no bypass was detected.

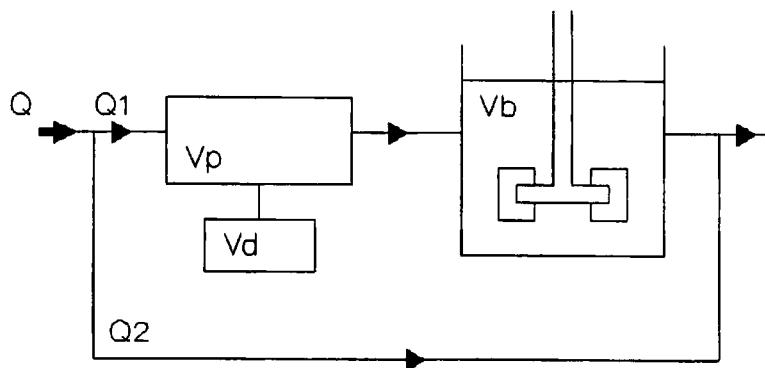


FIGURE 11

Representation of the multiparameter non-ideal flow model after Levenspiel. Reprinted from ref. (69).

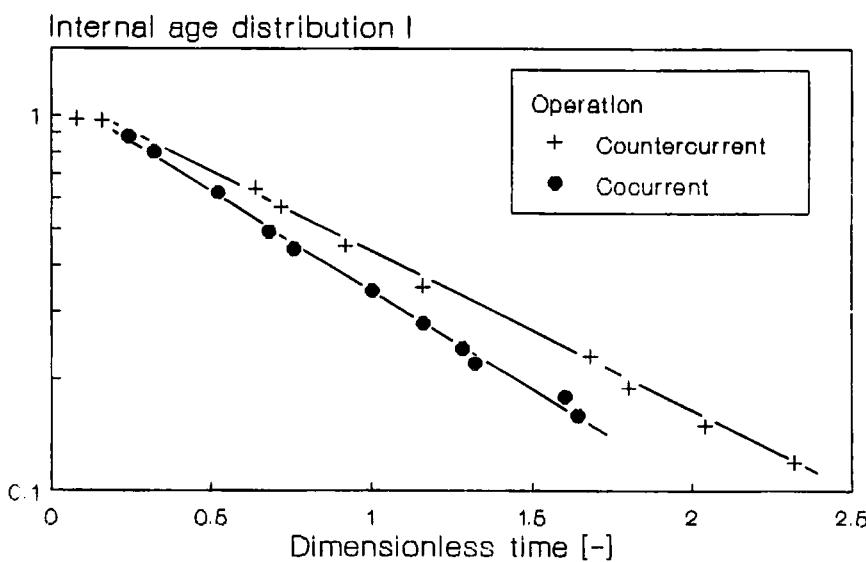


FIGURE 12

Internal age distribution function of the effluent during electrolytic flotation. Reprinted from ref. (70).

The kinetics of the process are usually thought to be first-order³. Under steady-state conditions, a mass balance for the flotation cell may be used to describe the continuous process :

$$r = Q_{in} C_1 - Q_{out} C = k_1 C V \quad (8)$$

where r is the overall flotation rate, Q is the volumetric flowrate, C_1 and C are the inlet and exit concentrations, respectively, and k_1 is the flotation rate constant; the latter is really a complex function of various parameters, e.g. the particle and bubble size, the particle characteristics, the concentration of collector etc.

The left hand side of this equation represents the mass balance between the flow going in the vessel and that flowing out, whereas the right hand side represents the flotation rate. There is an inherent assumption that the gas bubbles will carry the particulate matter at the top, while no liquid is lost, i.e. $Q_{in} = Q_{out}$. The mean residence time (τ) is

$$\tau = \frac{V}{Q} \quad (9)$$

and, using this, the equation may be rearranged as follows :

$$C = \frac{C_1}{1 + k_1 \tau} \quad (10)$$

This equation may then be used to predict, with reasonable approximation, the concentration of a particular species in the effluent flowing out of the flotation cell, as a function of the feed concentration, the flotation rate constant and the mean residence time. Thus, many calculations may be performed, e.g. if the outlet concentration is expected to be above that imposed by a local water authority, it is possible to use eqn. (10) to determine the mean residence time of the effluent and from this the adjusted volumetric flowrate.

Figures 13 and 14 present results obtained with the electrolytic flotation of a paint effluent and an emulsified oil/water dispersion. Deviations were observed at high throughputs or high feed concentrations, showing possibly an upper limit in flotation operation. It has also been found that there is an optimum concentration, where the rate reaches a maximum⁷².

If such deviations from first-order kinetics occur, the order may be computed by fitting experimentally-obtained data about the amount of material (m) floated in a given time (t_M), using the following equations⁷³ : for $n = 1$,

$$\ln\left(\frac{1}{1 - m}\right) = K t_M$$

and for $n \neq 1$

$$\frac{1}{n-1} [(1-m)^{1-n} - 1] = K t_M C_0^{n-1}$$

where C_0 is the concentration at time $t = 0$. This has been successfully applied to the flotation of magnesium carbonate fines⁷³.

Flotation kinetics is presently approached through the application of various models to results obtained mostly in laboratories, but also in pilot-plants as well as in industrial

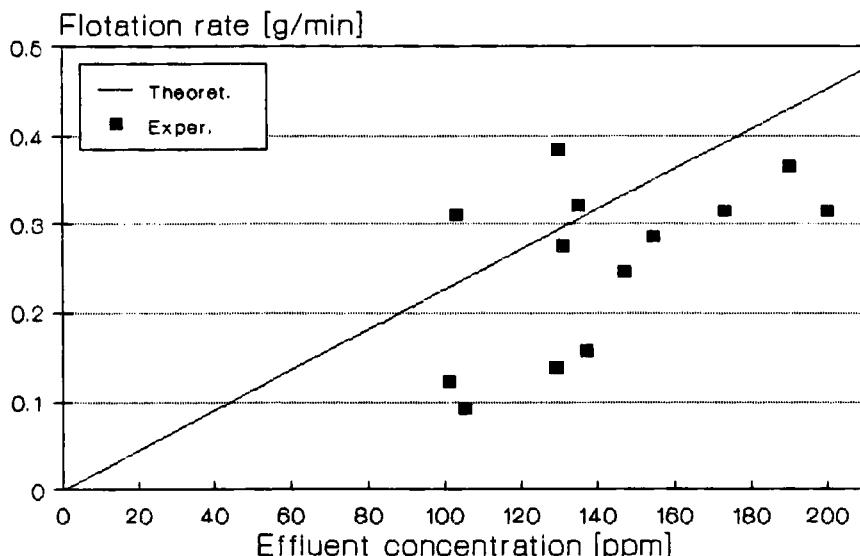


FIGURE 13
Continuous-mode electrolytic flotation of paint suspension. Reprinted from ref. (71).

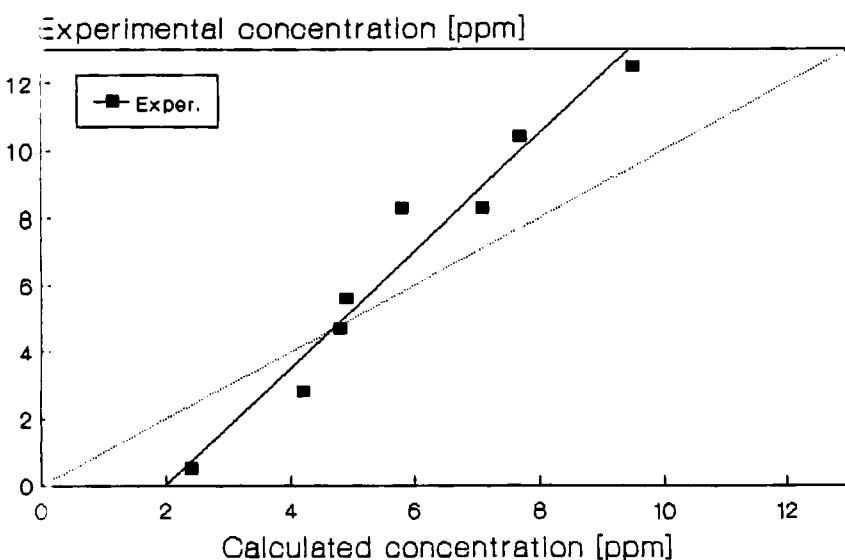


FIGURE 14
Experimental vs. calculated concentration of emulsified o/w effluent during electrolytic flotation. Reprinted from ref. (71).

TABLE V
A selection of flotation kinetics models^{74*}.

Model 1	$R = R^*(1 - e^{-k_1 t})$	Classical first-order model, where R is the recovery at time t and R* is the ultimate recovery for an infinite time
Model 2	$R = R^*[1 - \frac{1 - e^{-k_2 t}}{k_2 t}]$	First-order model with rectangular distribution of floatabilities
Model 5	$R = R^*[1 - \frac{1}{1 + \frac{t}{k_5}}]$	Fully mixed reactor model
Model 6	$R = \frac{k_6 t}{(1 + k_6 t)^m}$	Gas/solid adsorption model
Model 9	$R = R^*[1 - \frac{\ln(1 + k_9 t)}{k_9 t}]$	Second-order model with rectangular distribution of floatabilities
Model 10	$R = (1-z)(1 - e^{-k_{10f} t}) + z(1 - e^{-k_{10s} t})$	Three-parameter model, fast (f) and slow (s) floating components
Model 13	$R = R^*[1 - (\frac{a}{a + t})^p]$	Three-parameter model (gamma distribution)

* The author's numbering has been kept.

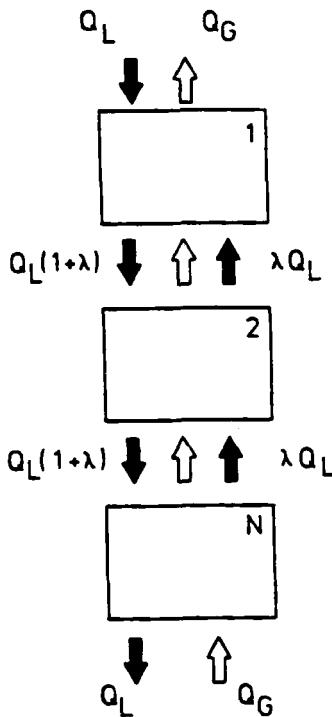


FIGURE 15

Model of N -zones-in-series for the representation of the flotation column. Reprinted with permission from ref. (47); copyright Elsevier.

operations. Table V presents some of these models - an extensive review of flotation kinetics has been recently published⁷⁴.

A more complex model has been used to describe the liquid-phase mixing in a flotation column⁴⁷, involving a fully-mixed zones-in-series model which allows for both forward and backward flow between the zones (Figure 15). Thus, the model involves two parameters : the number of zones and the ratio of backwards to net forward liquid flow. Using experimental results (Figure 16), a set of feasible cell numbers - flow ratios was determined; more experimental results at different gas and pulp flow conditions were then used to determine the optimum pair of parameter values.

The degree of mixing in the gas-liquid flotation column, which is of major importance for its performance, was found to be clearly affected by the relative magnitude of the gas and liquid flowrates, among other parameters. The increase of the gas flowrate was found to increase the degree of mixing in the column, whereas the liquid flowrate increase had the opposite effect.

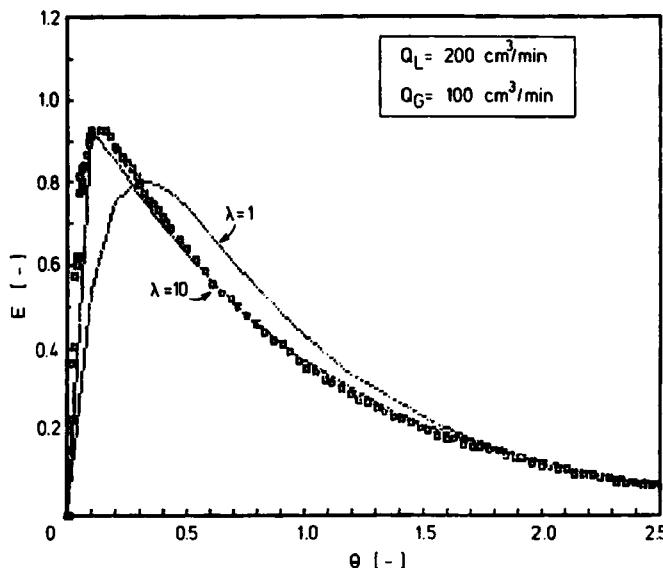


FIGURE 16

Modelling of liquid-phase mixing in a flotation column: fitting a theoretical to the experimental residence time distribution curve. Reprinted with permission from ref. (47); copyright Elsevier.

Phenomena taking place in the froth are much more difficult to describe. A model separating phenomena taking place in the pulp from those occurring in the froth, assumed that the latter was perfectly mixed⁶⁴; this assumption has not been widely accepted since some postulate that this is a plug flow zone. The resulting models tend to be cumbersome and parameters difficult to quantify.

Finally, extensive development has taken place in the '80s on the design and implementation of computer programs to simulate flotation circuits^{2,75}.

8. FLOTATION IN MINERAL PROCESSING

Flotation has found its main application in mineral processing (termed froth flotation). In mineral processing, it is the coarser size that is most important, for obvious economic reasons⁷⁶, as the fines are usually discarded in practice.

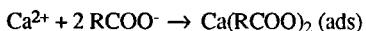
It is not really possible to cover in a single chapter all its aspects; excellent review books exist (see refs. 2, 11, 20, among others). However, a short description of this field will be included for the sake of completeness; the processing of magnesium carbonates will be used as an illustration of minerals flotation studies.

Salt-type minerals have the general type $M_X A_Y$, where M is some cation, e.g. Mg^{2+} , Ca^{2+} , Bi^{2+} , and A is usually an anion, e.g. F^- , CO_3^{2-} , SO_4^{2-} , WO_4^{2-} , PO_4^{3-} etc. These minerals are characterized by a slight dissolution in aqueous dispersions, where they give metal ions, like calcium and magnesium, and hydrolysis products. Fatty acids are often used as collectors; they adsorb on the minerals (Figure 17), depending generally on the conditions, either by physical or chemical adsorption or both, with chemisorption predominant in the alkaline pH region⁷⁷. Fatty acids and their salts in aqueous solutions are also known to be hydrolysed and the concentration of different species is a function of pH as well as total concentration (Figure 18).

Experiments were performed with magnesium carbonates (dolomite and magnesite)⁵⁹. Experimental evidence suggested that the raising of pH would cause a rapid decrease in the solubility of the $CaCO_3$ component of dolomite; a much stronger affinity of oleate ions towards Ca rather than Mg ions was also displayed.

One of the parameters investigated was the influence of conditioning time on pH (with or without the presence of other reagents); the results are shown in Figure 18 for dolomite. When the collector (sodium oleate) was present, the final pH was changed to 9.5. This was attributed to the capture of Ca^{2+} in solution by oleate ions, resulting in more Ca^{2+} , Mg^{2+} and CO_3^{2-} ions being released from the surface into the solution. Service water certainly contains significant amounts of calcium and magnesium ions, which are considered detrimental in flotation with fatty acids³⁵.

Another parameter of major importance¹¹ was the pH : experimental results obtained with dolomite are illustrated in Figure 19. At intermediate collector concentrations (10-40 ppm) maxima and minima of dolomite recovery were observed. It has been suggested that with a collector concentration of 20 ppm, the principal mechanism for total dolomite flotation at pH 7.0 was readorption of dissolved calcium in the form of calcium dioleate molecules, through a process of hydrophobic aggregation :



(with K_{sp} of calcium dioleate $10^{-15.4}$ - ref. 78). The experimental results, obtained in a modified Hallimond tube⁵⁹, suggested that, at pH 7.0, a major transfer of calcium from dolomite surfaces to magnesite surfaces occurs during the flotation of a mixed mineral feed. At pH 11.0, on the other hand, the suggested mechanism was conventional adsorption of oleate ions onto surface calcium sites following the suppression of $CaCO_3$ solubility to the point at which most of the calcium remains on the dolomite surface layers.

Between these points lies a zone of intermediate pH values, where with increasing alkalinity the dolomite recovery declines to a minimum before recovering sharply as the pH approaches 11.0 (Figure 19). These inferior intermediate-zone results could probably be regarded as reflecting varying degrees of permanent net loss of calcium from the dolomite surfaces. The principal reason for such losses is likely to be the change in solution equilibrium between carbonate and bicarbonate ions with increasing alkalinity.

Preferential adsorption of oleate to the dolomite should also be favored by the ζ -potential gap (see, for example, Figure 9), if these curves remain valid for a mineral mixture. Although the solubility of $CaCO_3$ is very sharply reduced at pH 11.0, a certain amount of dissolution still takes place.

The floatability of a solid mineral, under certain conditions - for instance, pH, concentration of collectors, modifiers etc. - can be often examined by contact-angle measurements, although there is nowadays some kind of dispute over the way data from such experimentation may be

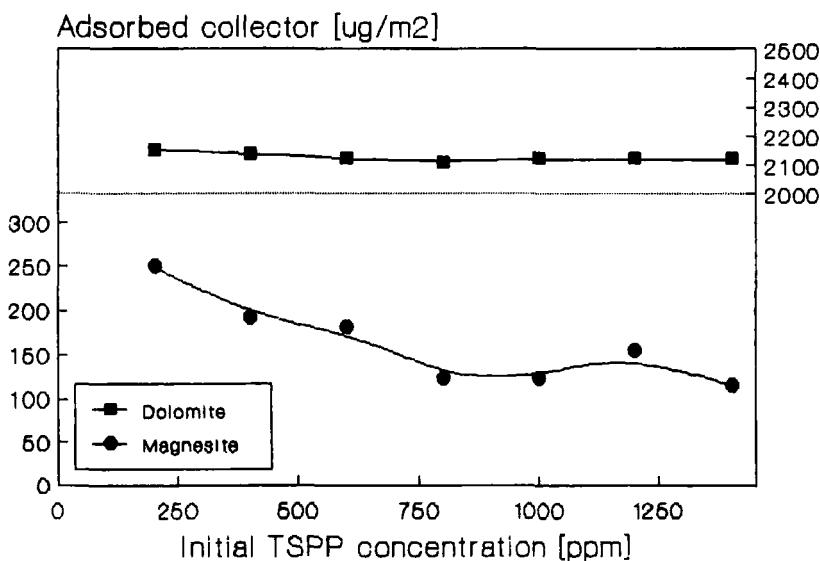


FIGURE 17

Effect of the amount of tetrasodium pyrophosphate (TSPP) added on the adsorption of fatty acid by fine mineral particles. pH 11.0; initial collector concentration (tall oil fatty acid) 40 ppm. Reprinted with permission from ref. (39); copyright Science and Technology Letters.

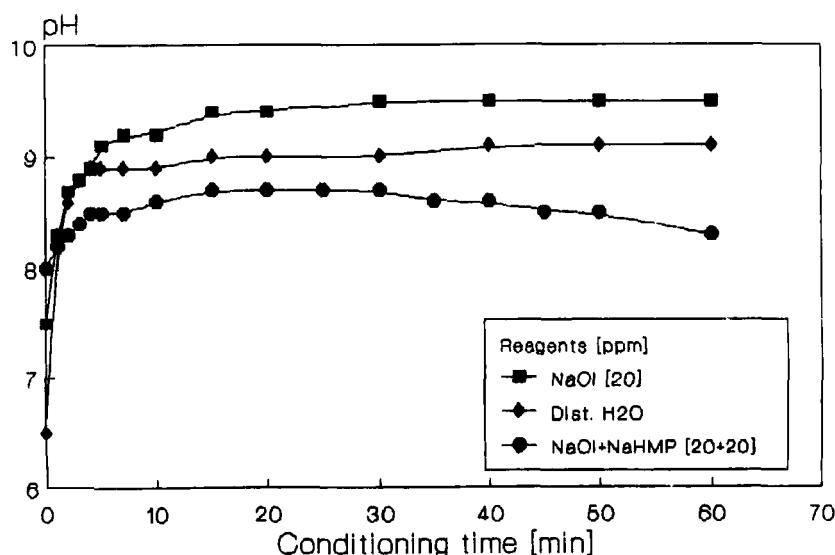


FIGURE 18

Effect of conditioning time on the pH of dolomite suspensions. Reagents used: sodium oleate (NaOl) and sodium hexametaphosphate (NaHMP). Reprinted with permission from ref. (35); copyright Elsevier.

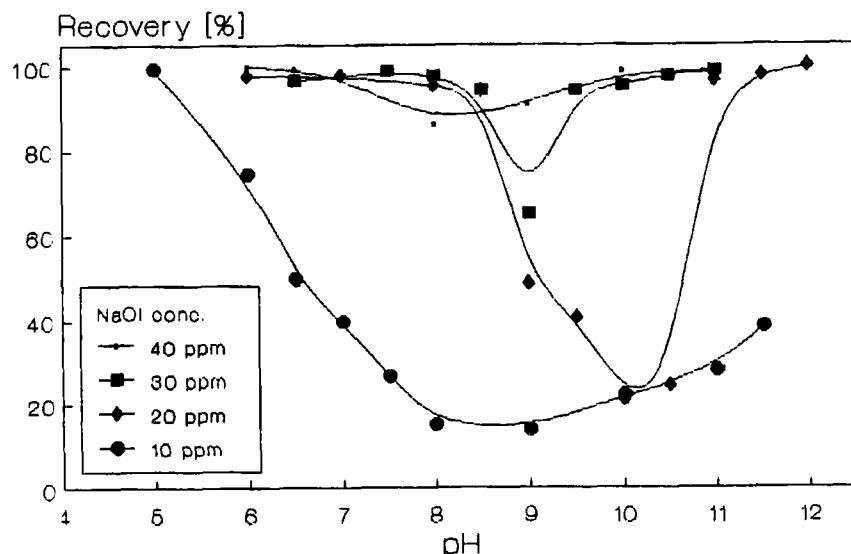


FIGURE 19

Effect of pH on froth flotation of dolomite for various collector concentrations (sodium oleate). Reprinted with permission from ref. (59); copyright IMM (UK).

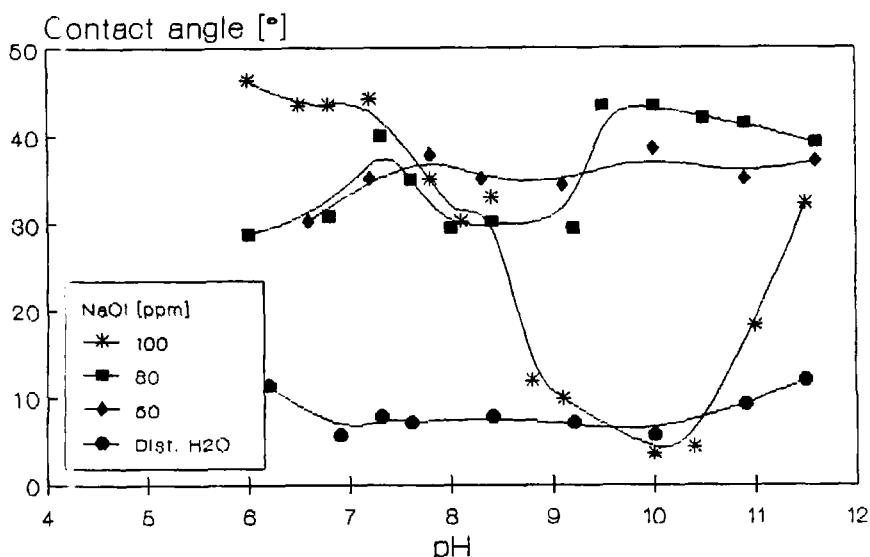


FIGURE 20

Contact-angle measurements of magnesite at various pH and collector (sodium oleate) concentrations. Reprinted with permission from ref. (59); copyright IMM (UK).

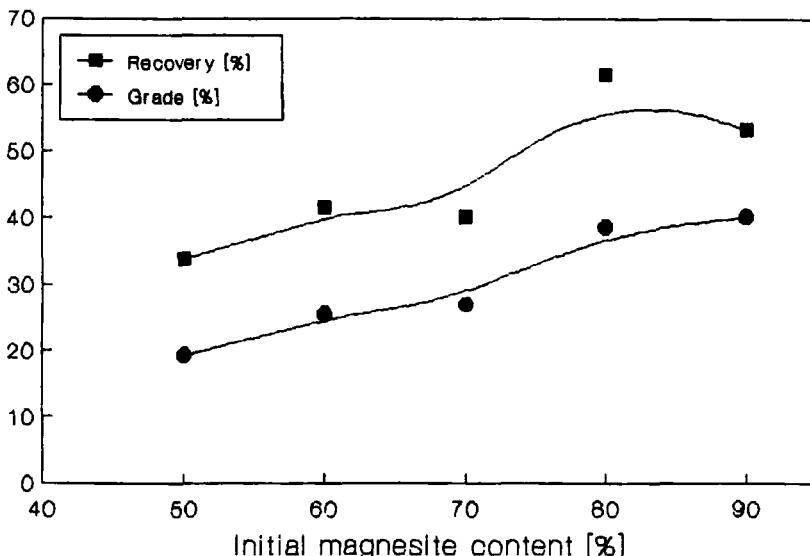


FIGURE 21

Influence of initial magnesite content of binary magnesite / dolomite mixtures on the magnesite grade and recovery in froth flotation. pH 11.0. Reagents used : [sodium oleate] = 20 ppm, [sodium silicate] = 240 ppm. Reprinted with permission from ref. (35); copyright Elsevier.

evaluated⁷⁹ in order to assess correctly the hydrophobicity of the mineral. Figure 20 shows the measurements taken with magnesite.

One of the most important features of flotation in mineral processing is the selectivity attained in the separation process; this is a fundamental difference from effluent treatment, where a bulk removal of solids is expected. In order to achieve this selectivity, modifiers, either organic or inorganic, are often used.

Magnesite and dolomite are both minerals which contain a common cation (magnesium). Salt-type minerals present similar behavior during flotation, due mainly to the similar chemical composition of their surface and the great interphase activity of the flotation collectors, that are usually applied. For this reason, the separation of this type of minerals from others of the same type is quite difficult.

Selective separation by flotation of artificial mixtures of magnesium carbonate minerals was investigated with or without modifiers^{35,59,80}. Sodium silicate was used as a modifier²³; it is known that salts of this modifier, e.g. $\text{CaSiO}(\text{OH})_3^+$, are formed either on the particle surface by chemisorption or in solution and are then adsorbed on the surface, depending upon the prevailing conditions (pH, concentration). Figure 21 shows typical results of the grade and recoveries achieved. The concentrate was enriched in magnesite for initial contents of magnesite in the mixture of 50-80%. In experiments with a single mineral⁸⁰, however, it was observed that sodium silicate did not affect the recovery of magnesite (as a function of pH), but was only causing a slight activation of the order of 15%.

9. CONCLUDING REMARKS

Very large flotation units have been or are being installed nowadays, demonstrating the reliability and success of the process. It has become indispensable for mineral processing plants; without it, the availability and the price of many non ferrous metals - like copper, lead, zinc, molybdenum etc. - would be totally different from what it is now. In wastewater treatment plants, flotation is being used to a lesser extent, so far. The analysis of the kinetic data available has also been successful, permitting the development of computer programs which, in conjunction with on-stream-analysis, are able to control flotation plants.

There are several areas in froth flotation, where more work is needed : cells (both mechanical and columns) have been designed, so far, on a more or less empirical basis, starting by building a cell and then trying to optimize it by retrofitting. Only lately work has started into the investigation of the phenomena occurring in flotation cells, in order to achieve cell design and optimization from first principles. These include :

- the physico-chemical phenomena related to the dispersion of species in the solution, the transfer from solution to the surface of bubbles and particles etc. Of particular interest is flocculation of fine particles; a step further in this field would be the achievement of selective flocculation, using specific reagents (used so far on a much smaller scale in analytical chemistry, while preconcentrating samples).
- the mixing of the phases in the cell. This is a field where most of the knowledge has come from chemical reactors, with a distinctly different configuration (rotor design and position, cell geometry, gas and liquid flowrates etc.). The cell features must be investigated specifically for the flotation cell : effects of turbulence on particle-bubble collision, adhesion and detachment; the optimization of the cell (in terms of power and the gas flowrate required both for suspending the particles and floating them in mechanical cells);
- the bubble generation apparatus; finer bubbles (less than about 130 μm) will increase the performance of the cell, especially in systems with fine particulate matter, e.g. flocs, droplets etc.;
- collectorless flotation (electrolytic flotation has been successful, so far, in this field);
- application of new conditioning techniques (e.g. bioleaching), in order to improve liberation and recovery;
- finally, application of the process in other fields : oily wastes (refinery effluents), treatment of wastewater for potable use, separation of fine chemicals produced in large-volume batches.

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11. NOMENCLATURE

- A : froth area
C, C_0 , C_1 : concentration
 d_b : bubble diameter
 d_p : particle diameter
D : impeller diameter

F	: F-curve (response to a step impulse)
g	: gravitational acceleration
H	: cell height
I	: age distribution function
k,k ₁	: flotation rate constant
K	: constant
L	: cell length
m	: amount of material
n	: order of flotation kinetics equation
N	: agitator rotation speed
N _{Fr}	: Froude number
N _P	: power number
N _{QA}	: air flow number
N _{QL}	: impeller discharge number
N _{Re}	: Reynolds number
N _{We}	: Weber number
P	: power consumption
Q, Q _A , Q _L , Q ₁ , Q _{in} , Q _{out}	: flowrate
r	: flotation rate
R	: impeller aspect ratio
t _M	: flotation time
V	: total volume of cell
V _D	: dead volume
V _B	: backmixed flow volume
V _P	: plug flow volume
W	: cell width

Greek letters

θ	: dimensionless time
μ	: viscosity
ρ	: specific density
σ	: surface tension
τ	: mean residence time

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