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DEVELOPMENT OF NOVEL FLOTATION-ELUTRIATION METHOD FOR COARSE PHOSPHATE BENEFICIATION

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DEVELOPMENT OF NOVEL FLOTATION-ELUTRIATION METHOD FOR COARSE PHOSPHATE BENEFICIATION

FINAL REPORT

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PERSPECTIVE

H. El-Shall, Project Manager
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In the beneficiation of Florida phosphate rock, flotation plays a predominant role because it is the most economical way to separate the phosphate values from the sand and other impurities contained in the matrix. Typically, the matrix is washed and deslimed at 150 mesh. The material finer than 150 mesh is pumped to clay settling ponds. An estimate of 20-30% of the phosphate (contained in the matrix) is lost with these clays. The rock coarser than 150 mesh is screened to separate pebbles (-3 l/4 + 14 mesh) which are of high phosphate content. Washed rock (-14 mesh + 150 mesh) is sized into a fine (usually 35 x 150 mesh) and a coarse flotation feeds (usually 14 x 35 mesh) which are treated in separate circuits.

Flotation of phosphates from the fine feed (35 x 150 mesh) presents very few difficulties and recoveries in excess of 90% are commonly achieved using conventional mechanical cells.

On the other hand, recovery of the coarse phosphate feed is much more difficult and flotation by itself normally yields recoveries of just 60% or even less. In the past, hammer mills were used for size reduction of the coarse feed. However, due to high maintenance costs and loss of phosphates as fines (generated during milling) its use has been discontinued. The industry, however, has taken other approaches to circumvent the problem of low floatability of coarse particles. For instance, such approaches are exemplified by the use of gravitational devices such as spirals, tables, launders, sluices and belt conveyors modified to perform a "skin flotation" of the reagentized pulp. Although a variable degree of success is obtained with these methods, they have to be normally supplemented by scavenger flotation. In addition, some of them require excessive maintenance, have low capacity or high operating costs. Their performance is less than satisfactory and in certain cases their use has been discontinued.

The exact reasons that explain the low recoveries of coarse particles in conventional flotation machines are not clear. Nevertheless, there have been several hypotheses about the behavior of coarse particles. For instance, the low floatability of large particles could be related to the extra weight that has to be lifted to the surface (usually under highly turbulent conditions) and then transferred and maintained in the froth layer. Factors such as density of the solid, turbulence, stability and height of the froth layer, tenacity of the particle - bubble attachment, depth of the water column, viscosity of the froth layer, and other variables that can indirectly influence these factors determine the floatability of coarse particles.

Some effort towards improving the flotation of coarse particles through stabilization of the froth layer, increase in the froth height, improvement in froth viscosity. etc., through the addition of fine particles and/or different frothers have been undertaken. In this regard, beneficial results have been obtained by modifying the flotation chemistry in coal and sulfides industry. To test this concept in the phosphate flotation, FIPR granted funds (FIPR #87-02-067) to University of Florida. The results from both laboratory and plant scale testing are encouraging. The final report is ready for publication and distribution to interested parties.

Changes in the design of the flotation cells presently used in the industry could constitute an alternative route to attack the coarse flotation problem. This constitutes the major objective of this project.

The specific goals of this project as stated in the original proposal were to establish the reasons that explain the low recovery of coarse particles in conventional flotation, and on this basis, to design and evaluate at the laboratory level a new type of cell for coarse particle beneficiation. After the first year of work substantial progress was achieved and both objectives were partially accomplished. The main conclusion obtained from the theoretical and experimental study developed during the first year was that turbulence in mechanical cells is the most important limitation to coarse particles recovery. It was demonstrated that recovery in conventional cells decreases as agitation speed increases beyond a certain critical value. The other major limitation to coarse particles recovery is related to froth aspects such as: froth stability, transfer and stability of heavy particles in the froth. It was demonstrated that the larger the particle size the lower the recovery in the froth bed. Therefore, by increasing the froth depth, recovery of coarse values decreases.

Laboratory scale experiments showed that a column cell with a net upwards flow of water can take care of both limitations and therefore recoveries of up to 99% of phosphate up to 14 mesh is possible.

In view of the rapid advances during the first year, objectives for the second year were extended to include scale up and actual testing of a pilot size column. This latter objective was accomplished with the successful testing of a one foot diameter column at Noralyn plant of IMC during the first two weeks of April 1990. Results of this pilot test largely confirmed suggestions based on the laboratory work and demonstrated the soundness of the laboratory testing and the scale up procedure.

Results obtained were extremely encouraging. Up to 99% BPL recoveries were obtained with acceptable concentrate grade. The feed quality changed drastically during testing, but performance of the column was always much superior to that of the spirals circuit. Column recoveries were normally equal or better than the over all recovery obtained by the spirals plus the scavenger flotation combined.

Future research work that would make it possible to extend the conclusions of this report to a continuous operation as pointed out by the author should be focused on two aspects: continuous operation of the sparger, and automatic control of the tailings flow rate (to keep a constant concentrate flow rate). The performance of the filter cloth sparger seems adequate but this should be corroborated in a continuous operation. Automatic control of the tailings flow rate by a pinch valve coupled to a conductivity sensor has given encouraging results at Laval University, but this or similar approached should be tested in continuous operation under actual plant conditions.

Another suggestion for future research is the use of this type of cell for the flotation of dolomite using any of the various reagent schemes proposed in the past. Results discussed in this report suggest that column flotation of dolomite should yield higher recoveries than conventional cells, particularly in the case of anionic flotation at acidic PH values that are difficult to obtain in an agitated pulp containing carbonate minerals, Also, column flotation to upgrade low quality pebble could be attempted, this could be done with a minimum of grinding since column flotation of millimetre size particles seems possible. Pilot testing of these ideas should be encouraged. It seems that such recommendations for future work are sound and should be considered.

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Finally, I want to acknowledge Dr. H. El Shall of FIPR for the interest and welcoming reception he has accorded this project since its inception.

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EXECUTIVE SUMMARY

This report covers experimental results and discussion of the test work developed throughout this two year project that officially ended in July 1990.

Material is organized in three chapters. The most relevant experimental data obtained at laboratory level is presented in Chapter 1. Further details of this first laboratory phase can be found in Chapter 3 that includes all the work developed during the first year of the project that was not included in Chapter 1. Results obtained during the pilot test are presented in detail in Chapter II. Also in this Chapter a discussion of the scale up procedure, a description of the aerator and the column itself, and an analysis of the results are presented.

LABORATORY TESTS

The specific goals of this project as stated in the original proposal were to establish the reasons that explain the low recovery of coarse particles in conventional flotation, and on this basis, to design and evaluate at the laboratory level a new type of cell for coarse particle beneficiation. After the first year of work substantial progress was achieved and both objectives were at least partially accomplished. The main conclusion obtained from the theoretical and experimental study developed during the first year was that turbulence in mechanical cells is the most important limitation to coarse particles recovery. It was demonstrated that recovery in conventional cells decreases as agitation speed increases beyond a certain critical value. The other major limitation to coarse particles recovery is related to froth aspects such as: froth stability, transfer and stability of heavy particles in the froth. It was demonstrated that the larger the particle size the lower the recovery in the froth bed. Therefore, by increasing the froth depth, recovery of coarse values decreases.

Laboratory scale experiments showed that a column cell with a net upwards flow of water can take care of both limitations and therefore recoveries of up to 99% of phosphate up to 14 mesh is possible. Tests were performed under conditions as realistic as permitted by laboratory restrictions. (The terms “elutriation column,” “counter current column” and “negative bias column” are used interchangeably in this report to indicate a net upwards flow of water). In a negative bias condition the column is always full of liquid and no froth is formed on the surface. In general, column type cells make it possible to perform the flotation of coarse feeds in a low turbulence environment. Therefore, any type of column should yield higher recoveries than those obtained in agitated devices, but a thick froth layer has to be avoided. The introduction of an upwards water flow (usually termed a negative bias) is essential to reduce or eliminate the froth layer. Extensive testing at laboratory level with different coarse phosphate feeds indicated that it is possible to achieve a 98-99% recovery with reasonably good selectivity when up to 14 mesh phosphate particles are floated. Under comparable conditions recovery of the coarser phosphate fractions in conventional laboratory cells was considerably lower.

Column flotation was strongly dependent on the reagent level. The best results were obtained with total reagent dosages (tall oil + fuel oil) in the range 3.0 to 3.8 kg/ton. These figures correspond to the bulk amount of industrial grade reagents added without correcting for impurities or "active basis" or other considerations. During flotation itself, the most important operating variables were those related to bubble size. A small amount of frother (20-30 g/ton) was added to control bubble size. Contrary to our preliminary expectations we found that small bubbles (0.5-0.8 mm) yield far better recoveries than larger bubbles. Using bubble sizes larger than one millimetre decreased recovery sharply. A proper bubble size was easily achieved by using porous steel, ceramic or glass spargers in the presence of a small amount-of frother. A less sophisticated but equally effective sparger made with commercial quality filter cloth was adopted for the laboratory tests and also for the pilot column. Pore size and total surface of the sparger, frother concentration and air flow rate have an influence on bubble size.

Residence time of coarse phosphate particles in the laboratory column was determined to be just a few seconds, but this was enough to achieve very high recoveries. Such results indicate an extremely fast flotation kinetics. Capacity estimations based on these laboratory tests indicate about 8 tph/m^2 (0.7 tph/sq.feet) or about 7 tph/m^3 cell (0.2 tph/ cubic feet).

PILOT TEST

In view of the rapid advances made during the first year, objectives for the second year were extended to include scale up and actual testing of a pilot size column. This latter objective was accomplished with the successful testing of a one foot diameter column at Noralyn plant of IMC during the first two weeks of April 1990. Results of this pilot test largely confirm suggestions based on the laboratory work and demonstrate the soundness of the laboratory testing and the scale up procedure.

The pilot column featured a new aerator design that has a number of advantages over other conventional aerators: better distribution of bubbles, easy to replace or-inspect without shouting down the column, and low cost. Column dimensions were 1.0 foot diameter by 5.5 feet height, with an additional section that can increase the total height to 8 feet. This column was installed in the spirals section of IMC's Noralyn plant and fed by gravity from one of the feed distributors at a rate of 0.8 to 1.4 tph (metric tonnes are used throughout this report). According to information provided by IMC, collector dosage added to the spiral feed during testing was 3.7 and 4.0 lbs per tonne (of concentrate) of fuel oil and soap respectively. The only reagent added to the column was a small amount of frother in the water introduced at the bottom of the column.

Results obtained were extremely encouraging. Up to 99% BPL recoveries were obtained with acceptable concentrate grade. The feed quality changed drastically during testing, but performance of the column was always much superior to that of the spirals circuit. Column recoveries were normally equal or better than the over all recovery obtained by the spirals plus the scavenger flotation combined.

The effect of several operating variables was studied during the pilot test: frother concentration, air flow rate, water flow rate, feed rate, sparger surface and residence time . The most critical variables were, as in the laboratory testing, those related to bubble size. The presence of a frother was essential to achieve the fine bubble size distribution necessary for a good recovery. Dosages of 30-60 g/ton of polyglycol type frothers proved to be quite effective. Aerator surface was also an important variable, a surface of at least half the column surface was required to obtain a proper bubble size.

Recovery increased with increasing air flow rates up to about 0.5 cm/s (about 20 lpm). Air flow rates higher than about 0.8 cm/s tended to reduce recovery. Concentrate grade was independent on air flow rate. Recovery also increased when water flow rate was increased, but at the expenses of lower grades. Optimum water flow rate was about 0.3 cm/s.

Flotation kinetics was extremely fast which is in agreement with the laboratory results. A collection zone just 3 feet long yielded recoveries of over 90 % BPL. The bulk of the recovery occurs in the first 50 cm of column. Optimum performance of the column was observed with about 0.8 tph (1.1 tph per square feet) but it is likely that capacity can be increased if column length is increased or if collector dosages and the oil/soap ratio are optimized.

These pilot test results suggest that a column to treat 50 tph of coarse phosphate feed should have the characteristics summarized in the table presented next. If this scale up projection is correct, it means that just two columns 6x6x8 feet each could do the job now done by a circuit having about 100 spirals plus conventional scavenger flotation.

Scale up of pilot test results to 50 tph capacity.

Column surface	3.35 m^2	(6 x 6 feet)
Column height	3.0 m^2	(10 feet)
Aerator surface	2 m^2	(21 square feet)
Air flowrate	1.5 m^3/min	(50 cfm)
Water flowrate	1.2 m^3/min	(300 gpm)
Recovery	+95% BPL	
Concentrate Grade	7-10% Insolubles	

I hope that this report helps to point out the valuable contribution that column type cells can make in phosphate beneficiation. Columns are gradually replacing conventional cells in many flotation operations. Most of the technical advantages that make columns a preferred type of cell in many modern operations are also present in phosphate beneficiation. Furthermore, the experimental work presented here shows that columns can provide the low-turbulence conditions that are necessary to avoid abrasion of the mineral surface and particle detachment.

Future research work that would make it possible to extend the conclusions of this report to a continuous operation should be focused on two aspects: continuous operation of the sparger, and automatic control of the tailings flow rate (to keep a constant concentrate flow rate). The performance of the filter cloth sparger seems adequate but this should be corroborated in a continuous operation. Automatic control of the tailings flow rate by a pinch valve coupled to a conductivity sensor has given encouraging results at Laval University, but this or similar approaches should be corroborated in continuous operation under actual plant conditions.

Another suggestion for future research is the use of this type of cell for the flotation of dolomite using any of the various reagent schemes proposed in the past. Results discussed in this report suggest that column flotation of dolomite should yield higher recoveries than conventional cells, particularly in the case of anionic flotation at acidic pH values that are difficult to obtain in an agitated pulp containing carbonate minerals. Also, column flotation to upgrade low quality pebble could be attempted, this could be done with a minimum of grinding since column flotation of millimetre size particles seems possible. Pilot testing of these ideas should be encouraged.

CHAPTER 1

LABORATORY TESTING

MATERIALS AND METHODS

Phosphate Ores.

Two samples of coarse phosphate ore of about 1400 pounds were obtained from IMC of Bartow, Florida. This material was analyzed and characterized and then divided into 1.0 kg batches suitable for laboratory testing. The coarser sample corresponds to the feed of the spiral concentration circuit at Noralyn plant. The other sample is a coarse flotation feed of the same plant. Particle size distribution of both materials is presented in Table 1. Note that the spiral feed is much coarser than normal coarse phosphate flotation feeds. Size by size analyses of both samples are presented in Table 2.

Both samples are industrial quality products and were used as received. They were subjected to no purification or other manipulation to assure that results obtained in this research could be directly related to industrial practice. Tap water was used for all testing involving phosphate minerals. Maximum water recycling as allowed by experimental constraints was the norm during column flotation tests. Chemical assays of the two phosphate samples indicated 30.5 % BPL and 58% insolubles for the coarse flotation feed and 44.5% BPL with 39 % insolubles for the spiral feed. Both samples contained essentially francolite and quartz with minor impurities of clays and feldspars.

Reagents

Some early exploratory tests reported here were carried out with technical grade reagents provided by different suppliers. Fatty acid (Acintol AF3) was obtained from Arizona Chemicals. All other test work was conducted with industrial grade samples of collectors and oil extenders used in Florida phosphate plants. These include fatty acids (Tall oil) soaps, diesel fuel, diesel oil, amine condensate, Armak 2HT-75 (a quaternary ammonium compound) and sodium silicate kindly provided by IMC Co. These reagents were also used “as received”.
Tap water of total hardness of about 100 ppm calcium carbonate was used throughout the experimental work.

Table 1.- Particle size distribution of phosphate samples.

	% Retained	
Mesh	Coarse Flotation Feed	Spiral Feed
14	0	1.9
16	0	6.3
20	2.6	13.3
28	17.0	43.6
35	29.0	20.2
48	27.2	9.8
65	15.2	3.3
-65	8.9	1.5

Table 2. Size analyses of phosphate samples.

Mesh	Coarse Flotation Feed		Spiral Feed	
	BPL %	Insols%	BPL %	Insols %
+ 20	-	-	61.8	16.7
20x28	45.0	38.7	48.5	34.2
28x35	31.8	56.7	34.5	52.9
35x48	24.1	67.5	23.1	66.5
- 48	23.5	67.7	22.0	70.4

Laboratory Column.

Considering the coarse size of the ore we assumed a plug type flow of particles and negligible vertical diffusion. Therefore dispersion of particles can be neglected and residence time would be a direct function of column length and independent on column diameter. While it seems necessary to increase the residence time of the particles i.e. increase column length as much as possible, at the same time it is necessary to reduce the height/diameter ratio of the laboratory column to a value in line with full size columns. These two criteria oppose each other so we decided to begin with a compromise ratio of about 12, and then to study experimentally the influence of the residence time on recovery.

A 7.0 cm internal diameter laboratory column was chosen because it is an adequate size to work with in the laboratory and experience with conventional columns indicate that 5.0 cm laboratory columns provide meaningful information for scale up to full size units. The scale up criteria were to keep constant the air and water velocities (i.e. constant flow rates per unit area of column); and to keep a constant ratio: sparger area to column area. For conventional columns it has been suggested a ratio of one or larger, however, for coarse particles flotation we think that given the low external surface area of the particles a lower ratio can be tolerated.

Pumps, aerator and feeding system were dimensioned according to the requirements of the 7 cm column. All pumps used were variable velocity peristaltic pumps which allowed a precise control of flow rates. The body of the column was a plexiglass tube divided into different modular sections that were bolted together and sealed with rubber seals. The upper section included a launder to collect the concentrate and the lower section contained the aerator and a spigot to introduce the bias water. Intermediate sections could be added as needed to have a given column length. Most tests were performed with a total column length of just 82 cm.

Some minor difficulties to design and built an adequate aerator were encountered but finally a simple and reliable air diffuser was built. It consisted of a 3.7 cm diameter 2.0 cm long tube with industrial filter cloth fixed at both ends. Total aerator surface is 21.5 cm which is still lower than the column area of 38.5 cm but a ratio sparger to column of about 0.6 seemed adequate at least for preliminary testing.

The feeding system presented a serious challenge due to the size of the particles, the relatively small amount of ore used, and to the absolute necessity of wet feeding and minimize abrasion. After several failed attempts we built a feeder based on a rotating spiral that allows reproducible feeding rates and showed no abrasion of the ore. Feed was introduced at a chosen depth and at the center of the column. A diagram of the column is presented in Figure 1.

Control instrumental incorporated to the column included:

l.- Automatic on line pH control system.
2.- Water flowmeter
3.- Air flowmeter and manometer
4.- Conductivity probes with automatic level control system.
5.- Hydrostatic pressure manometers for air hold up measurements.

Not all of this instrumental was used in every test. In most routine tests only the pH control system and the water and air flowmeters were used.

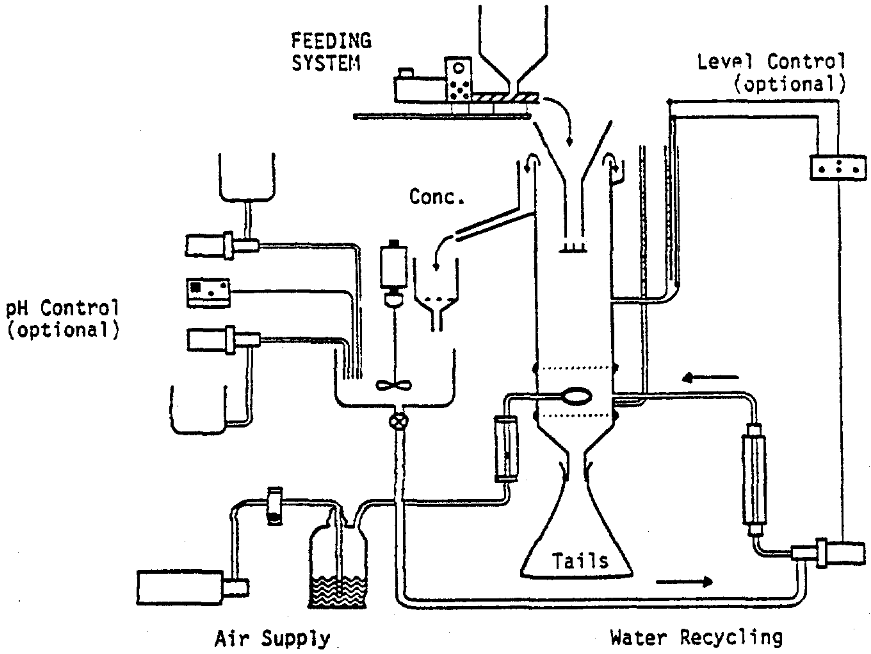

FIGURE 1 Diagram of Laboratory Column

Experimental Procedure.

Phosphate flotation tests were performed in a semi continuous mode with 1.0 kg samples. The ore was previously conditioned batchwise by tumbling with the desired amount of reagents during 8 minutes at 70% solids at pH 8.5-9.0. Before starting flotation, the conditioning solution was allowed to percolate to the column and then mixed with the water filling the column (tap water at pH 8.5-9.0 containing 30 mg/L aerofroth 65) and recirculated through the system during 5 minutes. This way the residual collector concentration and slimes content of the water used during flotation would be more representative of an actual continuous operation. During flotation a continuous feed rate of 200 g/min and a constant air and water flowrates were used. The concentrate was received in a 200 mesh sieve to allow immediate recycling of water. Volume of water used was that necessary to fill the column, tank and tubing, about 5.0 L in total. In most tests air flowrate was 1.5 L/min and water flowrate 1.0 L/min.

RESULTS

As could be expected flotation behaviour of phosphate was dependent on collector addition. Optimum collector dosage was found to be between 3.0 and 4.0 kg/ton. Very low recoveries were observed for total collector dosage below 3.0 kg/t, on the other hand, at dosages of about 3.6 kg/t recovery was better than 95%, at higher dosages limited gains in phosphate recovery were possible but at the cost of increased flotation of insolubles. Some typical results including mass balance calculations are shown in tables 3, 4, 5 and 6.

Table 3
Flotation of coarse flotation feed in column with 1.3 kg/ton tall oil and 2.3 kg/ton fuel oil. Frother 30 mg/l, air 1400 ml/min, water 1000 ml/min.

	Weight (grams)	BPL Recovery %	Grade BPL %	Grade Insols %
Concentrate	486	95.3	58.7	21.7
Tailings	498	4.7	2.82	96.2
Feed (calc)	984	100.0	30.4	59.8

Table 4. Flotation of mixture coarse+spiral feed.

(Same conditions as Table 3 but water flow reduced to 600 ml/min.)

	Weight (grams)	BPL Recovery %	Grade BPL %	Grade Insols %
Concentrate	472	96.7	60.8	18.9
Tailings	503	3.3	2.0	97.4
Feed (calc)	975	100.0	30.5	59.6

Table 5. Flotation of spiral feed (Same conditions as in Table 3)

	Weight (grams)	BPL Recovery %	Grade BPL %	Grade Insols %
Concentrate	621	97.8	68.2	9.0
Tailings	363	2.2	2.6	96.5
Feed (calc)	984	100.0	44.0	39.0

Table 6
Flotation of mixture spiral+coarse Feed. (Same conditions as in Table 3.)

	Weight (grams)	BPL Recovery %	Grade BPL %	Grade Insols %
Concentrate	557	96.7	61.1	18.5
Tailings	415	3.3	2.8	96.3
Feed (Calc)	972	100.0	36.2	51.0

As seen in these tables spiral feed recoveries higher than 95% are possible while eliminating about 85 % of the sand in a single rougher flotation. If the collector addition is increased, nearly 100 % recovery was achieved and about 60% of the sand was eliminated. For the coarse flotation feed recoveries were slightly lower with a maximum of 96-97% recovery at about 80% sand rejection, which yields a rougher concentrate grade of around 20% insols. Flotation of a mixture of both feeds yielded results intermediate to those when both feeds were floated separately.

A number of tests performed under various experimental conditions with both phosphate samples are summarized in Figure 2. Results fit nicely a grade versus recovery curve and indicate that very high recoveries with acceptable grades are possible. Also in this figure results obtained with a mixture of spiral and coarse flotation feed in a proportion 3:4 are presented. This is to simulate the response of the column to a non sized flotation feed or a poorly sized feed. Results show that the curve for the mixture falls in between the curves of the individual fractions. This indicates that the presence of the finer fractions does not affect to a large extent the flotation performance of the coarser particles.

These results (Figure 2 and also Tables 6 and 7) are quite encouraging because they show that the mixture can be floated as good as if the individual fractions are treated separately. This could be an important advantage over some traditional methods to beneficiate coarse phosphate fractions that are quite sensitive to the presence of fines. As compared to the normal treatment in separate circuits this alternative should result in considerable savings in sizing equipment while reducing complexity of the whole operation. Some of the concentrates and tailings of these tests were sized and analyzed to calculate recoveries and grades of the different size fractions.

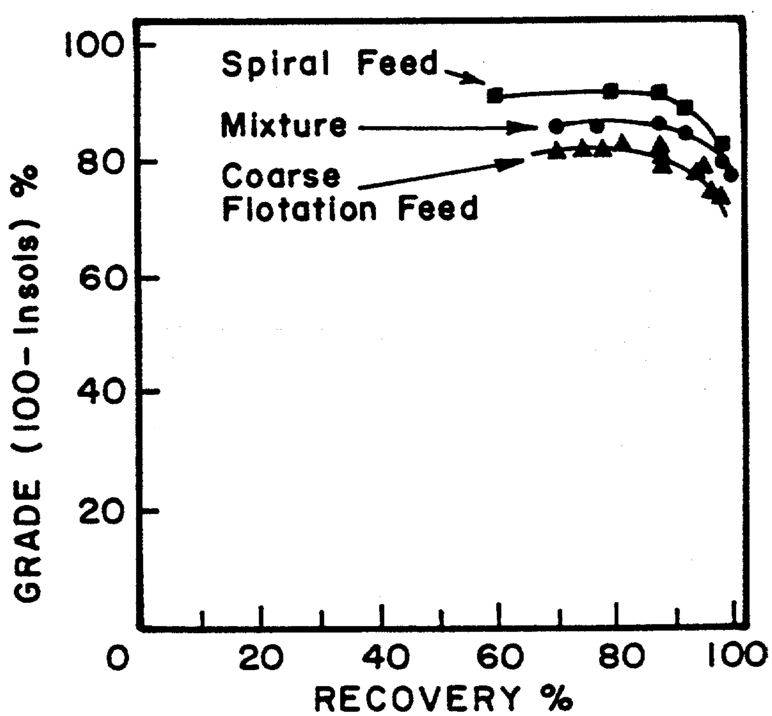

FIGURE 2. Grade/recovery curve for column flotation of phosphate.

Table 7.

Recovery and grade by size fraction for flotation of mixture coarse+spiral feed.

Mesh	Weight %	Grade % BPL Fraction	Grade % BPL Accum.	Recovery % Fraction	Recovery % Accum.
+ 28	13.3	70.1	70.1	14.5	14.5
28x35	52.7	70.0	70.0	57.1	71.6
35x65	25.4	62.0	67.8	24.4	96.1
-65	8.6	29.5	64.5	3.9	100

As shown in table 7 only the very fine sand floated and the plus 35 mesh concentrate which contains more than 70% of the phosphate values is practically market grade and does not need to be treated further. Therefore, it appears as an interesting alternative to perform a rougher flotation in a column before coarse sizing, and to send only the concentrate to coarse sizing. This way the plus 35 mesh fraction would be a final concentrate and only the minus 35 mesh rougher concentrate would have to be de oiled and cleaned in a cationic circuit. This scheme would have the advantages of eliminating the skin (spiral) flotation circuit, reducing to roughly one half the amount of material to be sized in the coarse circuit and reducing considerably the amount of rougher concentrate sent to de-oiling and to the cationic circuit.

Column versus mechanical cells.

Recoveries in the column were much higher than in parallel tests under similar conditions performed in conventional laboratory cells. In Tables 8 and 9 flotation results obtained with both phosphate samples in the column and in an Agitair laboratory cell are shown. Conditions for these test were as similar as permitted by the differences between the two apparatuses. Conditioning procedure, collectors, frother, pH and air flow were the same for both the column and the mechanical cell. Agitation speed in the mechanical cell was the minimum to avoid sedimentation of particles. As seen in Tables 8 and 9 while recoveries obtained in the column were higher than in the conventional cell, grade was maintained or even improved for the case of the finer feed. Tests with higher agitation speeds in the mechanical cell tended to decrease recovery and selectivity. Tests in a Denver laboratory cell gave slightly lower results than those obtained in the Agitair. Recoveries over 90% in both types of mechanical cells were possible when higher dosages of collector were added. However, the point we want to make here is not what maximum recovery can be obtained with the different apparatuses, but rather that with reagent conditions that yield only 50 to 60 percent recovery in mechanical cells, yield nearly 99% recovery in a counter current column.

Table 8

Flotation of spiral feed in column and in mechanical cell.

Collector 1 kg/t tall oil and 3 kg/t fuel oil. Air flowrate 1.4 L/min. Water flowrate 1.0 L/min. Values in parenthesis correspond to flotation the mechanical laboratory cell.

	Grams	BPL %	Insols %	Recovery %
Concentrate	667 (367)	62 (63)	18 (17)	99 (54)
Tailings	291 (604)	1 (33)	99 (56)	1 (46)
Calc. Feed	958 (971)	43 (44)	42 (41)	100 (100)

Table 9.

Flotation of coarse flotation feed in column and in mechanical cell.

Collector 1.5 kg/t tall oil and 2.5 kg/t fuel oil. Other conditions as shown in table 8.

	Grams	BPL %	Insols %	Recovery %
Concentrate	520 (356)	55 (52)	27 (31)	97 (59)
Tailings	447 (658)	2 (19)	97 (74)	3 (41)
Feed (calc.)	967 (1014)	31 (31)	59 (59)	100 (100)

Furthermore, flotation time in the mechanical cell was extended until the froth appeared to be free of floatable particles (between one and two minutes). In the column, however, flotation time was obviously limited to the residence time of the particles in the flotation zone. As shown later, residence time estimations obtained by timing free falling, collectorless phosphate particles in the column in conditions similar to the flotation tests, indicated an average value of 4.5 seconds for 14x20 mesh particles up to an average of 8.4 seconds for 35x48 mesh particles (for a 55 cm long flotation zone). Phosphate flotation in the column was, therefore, extremely fast, about one order of magnitude faster than in the conventional cell.

Effect of operating variables.

In addition to the effect of collector, recovery and grade obtained during column flotation depended to a lesser extent on factors such as water and air flowrates, frother concentration, bubble size, and depth of the feeding tube. In Table 10 the effect of bubble size on phosphate recovery is presented. It is clearly seen in this table that the smaller bubbles gave the best results. This is in line with theoretical and practical evidence that indicates that smaller bubbles have higher collision and attachment probability. However, in the case of large particles from theoretical calculations it was expected that in order to levitate heavy particles relatively large bubbles would be needed. During the flotation tests it appeared that small bubbles coalesced on the surface of hydrophobic particles to form the larger bubbles necessary for levitation.

Table 10. Effect of bubble size on spiral feed recovery.

Bubble Size (mm.)	Recovery %	Grade BPL %
1.6 (fritted glass)	32	69
1.1 (fine fritted glass)	88	67
0.8 (filter cloth)	94	66

In these experiments bubble size was modified by changing the aerator while keeping constant all the other variables, Bubble sizes were estimated by photography. It is clear that to optimize recovery it is necessary to decrease the bubble size. It was evident during the experimental work that bubble size was a complicated function of several parameters including pore size and total surface of aerator, air velocity, water velocity and frother concentration.

The effect of air velocity on phosphate recovery is shown in Table 11.(Air velocity is defined as the air flow rate in cm^3/s divided by the column surface in cm^2. It is a convenient way to express the air flow rate because is independent of the column diameter). These results came as a surprise because studies with pure quartz show that increasing air velocity in the region 0.3-1.3 cm/s results in higher recoveries which is in line with reported studies on the effect of air flowrate in conventional column flotation. However, as can be seen in Table 11 increasing air velocity decreases phosphate recovery. A possible explanation for this behaviour is that bubble size increases at higher air flowrates. Results presented in Tables 12 and 13 seem to support this hypothesis. As seen in Table 12 increasing frother concentration (i.e. reducing bubble size) increased recovery, although in this case, grade of the concentrate dropped rather sharply. In Table 13 the effect of aerator surface is shown. By increasing the total surface of the aerator, recovery increases for a given air flowrate.

Table 11.

Effect of air velocity on phosphate recovery.

Feed was a mixture of spiral and coarse flotation feed. Collector 1.2 kg/ton tall oil, 2.4 kg/ton fuel oil. Frother 30 mg/l.

Air Flowrate ml/min	Air velocity cm/s	Recovery %	Grade BPL %
1000	0.43	96.7	61.1
1500	0.65	89.3	60.0
2000	0.87	72.5	61.0
2500	1.08	65.2	51.5

Table 12.

Effect of frother concentration on phosphate flotation.

Air flowrate 2000 ml/min. Other conditions as in Table 11.

Frother (mg/l)	Recovery %	Grade BPL %
10	34.5	60.2
20	58.1	62.5
30	72.5	61.0
50	95.0	47.0
100	95.2	44.7

Table 13. Effect of aerator surface on recovery.

Aerator Surface (cm2)	Recovery %	Grade BPL %
22	72.5	61.0 (Air 2 lpm)
33	95.4	60.4 (Air 2 lpm)
22	65.2	51.5 (Air 2.5 lpm)
33	95.4	60. 8 (Air 2.5 lpm)

It was also evident that the bubble size determined the air hold up: the smaller the bubble size the larger the air hold up. This might be at least partially responsible for the effect of bubble size on recovery. It is obvious that this is an important issue and needs to be studied in more details. It appears on the other hand that smaller bubbles might be less selective, although this may only reflect the fact that approaching a 100% recovery implies the flotation of middlings and therefore increasing the amount of insols in the concentrate. Additional tests on the effect of bubble size are discussed later.

The addition of water was essential to avoid a froth layer on the top of the column. As shown in Table 14 if a froth layer was allowed to form, recovery dropped precipitously. Therefore, in order to float coarse phosphate particles, it is necessary to keep a negative bias (i.e. a net upwards flow of water) in the column to transfer the floated particles to the concentrate without the formation of a froth bed. Entrainment of gangue by the upwards flow of water was minimal and restricted to very fine particles. Water velocities used in column flotation were below 0.5 cm/s. (Water velocity is defined as the flow of water divided by the column surface). According to theoretical calculations this type of velocities should not elutriate particles larger than about 200 mesh.

Table 14 . Effect of froth thickness on spiral feed recovery.

Froth Depth (cm)	Water Velocity (cm/min)	Recovery %
0	15.6	98; 99; 98
0	0.0	95
1.0	0.0	56
2.0	0.0	30
4.0	0.0	0

This estimation was supported by entrainment experiments in which pure, fine quartz was "floated" in the column in the presence of frother but no collector. During actual column flotation of phosphate, entrainment of quartz particles larger than 0.1 mm was evident (see results in next section), but this entrainment was similar or even smaller than entrainment in conventional cells. This entrainment can be reduced by increasing the depth of the feeding tube and by reducing water velocity to the minimum necessary to avoid froth formation.

Cationic Flotation.

The term cationic flotation indicates here the use of cationic collectors to separate the quartz directly from the ore (not as usually referred to clean a rougher anionic concentrate). The non floated product is the phosphate concentrate and therefore recovery means the amount of phosphate remaining in the non floated fraction. In theoretical terms upgrading of coarse phosphate by this "reverse" flotation procedure present a number of advantages: sand is lighter than phosphate, particle size is smaller, particle shape is less spherical, and for the coarsest fractions sand represents the minor component of the feed. Therefore it is an alternative worth of study.
However the cationic route using azamine and diesel fuel gave rather disappointing results. If collector dosage is increased in order to float more quartz, fine phosphate starts to float and is lost in the froth product, but coarse quartz still stays in the cell contaminating the concentrate. Results obtained are summarized in Table 15. This poor performance of the cationic route was not a problem of the column itself but rather of the poor selectivity exhibited by the collector. Given its theoretical advantages it seems advisable to pursue this route with other collectors.

Results presented in the following section demonstrate that CTAB, a quaternary ammonium salt, is an excellent collector of pure quartz and that particles of up to 10 mesh can be recovered with moderate amounts of this collector. In view of this, some cationic flotation tests of the spiral feed using CTAB were performed. Results were a complete failure, sand did not float at all in the presence of phosphate. Addition of an extender and increasing the collector dosage to very high values did not change the situation. See Table 16.

Table 15.

Cationic flotation of spiral feed in laboratory column.

Air 1500 ml/min air and 1000 ml/min water. (Recovery refers to non floated phosphate). Frother 25 mg/l.

Test #	Azamine kg/t	Diesel fuel kg/t	Recovery %	Grade BPL%	Grade Insols %
16	1.0	1.0	54.5	61.8	18.2
17	1.5	1.5	28.8	62.1	18.5
18	1.0	1.0	73.2	60.5	19.3
19	0.7	0.7	95.6	46.5	37.2
20	0.9	0.9	94.0	49.2	34.1
21	0.9	0.9	85.2	56.1	25.4
22	0.7	1.35	84.5	53.1	29.2
23	0.5	1.5	90.8	50.1	34.0
24	0.6	1.2 *	98.8	45.6	39.0
25	0.8	1.2 *	98.4	46.2	38.5
26	1.2	1.8 *	98.1	52.4	35.2

* A specialty oil (Philflo oil) was added instead of diesel fuel.

Table 16. Flotation of spiral feed with CTAB under various conditions.

CONDITIONS	Insols(%)	Recovery (%)
250 g/ton, pH 8, Air 3000 ml/min	-	0
500 g/ton, pH 8, Air 3000 ml/min	-	0
1000 g/ton, pH 8, Air 3000 ml/min	65	5
1000 g/ton, ph 8, Air 1500 ml/min	54	4
600 g/ton, 1200 g/ton oil, pH and air as above.	73	3
1000 g/ton, 2000 g/ton oil, pH and air as above.	55	5

Under the conditions shown in Table 16 pure quartz of up to 10 mesh size floats nearly 100 percent. It was beyond the scope of this research to investigate the cause of this extremely different behaviour, but it is certainly a challenging problem that would be interesting to inspect in details because it could provide some clue to the difficulty encountered to float quartz particles from a phosphate feed.

A different type of cationic collector was tried on the basis of a recommendation by personnel from a phosphate plant. The collector (Armak 2HT-75) is also a quaternary ammonium compound but with a different hydrocarbon chain structure. On the basis of the same recommendation a specialty oil, Philflo oil from Phillips Petroleum, and an acidic conditioning procedure utilizing fluosilicic acid were utilized. Results were much better than with the other cationic collectors used. As shown in Table 17 reasonably upgrading and recovery were obtained, but performance is not as good as that obtained with the anionic route. However, the number of tests performed was quite limited and therefore it seems advisable to further explore this collector, particularly considering that the conditioning procedure seems to be quite important to get an efficient performance of this collector.

In any case results shown in Table 17 and 18 indicate that the column yields much better results than conventional cells.

Table 17.

Column flotation of spiral feed using 2HT-75 and Philflo oil.

Frother 30 mg/l, Air 1500 ml/min, water 1100 ml/min. Conditioning pH 4.5.

2HT-75 (g/ton)	oil (g/ton)	BPL Recovery %	Grade BPL %
1500	3000	18	70
1000	2000	22	70
500	1000	85	68
250	500	99	40
750	1500	48	67
600	1200	69	72

Table 18

Mechanical cell flotation of spiral feed with 2HT-75 and Philflo oil.
Conditions similar to Table 17.

2HT-75 (g/ton)	Oil (g/ton)	Recovery %	Grade BPL %
500	1000	100	40
1000	2000	99	40
1500	3000	85	56

BASIC RESEARCH

Effect of Agitation on Flotation.

For any given flotation system there are three critical impeller velocities that have to be satisfied simultaneously, namely: Critical velocity for particles suspension (Cvs), critical velocity for air dispersion (Cva) and critical velocity for bubble-particle detachment (Cvd). It is likely that air dispersion is a phenomena more or less independent on particle size. However, Cvs and Cvd should both depend on particle size. Intuitively, it can be predicted that larger particles would require a stronger agitation to keep the solids from settling i.e. a larger Cvs; on the contrary the value of Cvd would decrease because a heavier particle would detach more easily from a bubble than a smaller one.

With small particle sizes a rather wide range of impeller speeds exists where the agitation speed is large enough to exceed Cvs (and therefore fully suspend the particles) and at the same time agitation is low enough so that Cvd is not reached (and therefore bubble-particle aggregates are not destroyed). For this kind of particle size, flotation would present no problems as far as suspension and stability of aggregates is concerned. However, since Cvs increases with particle size and Cvd decreases, at a given particle size, Cvs will fatally surpass Cvd, and then, at no agitation speed both criteria can be met simultaneously.

Direct determination of Cvd in flotation machines is not possible. But at least the recovery of particles as a function of impeller speed and particle size can be measured. Flotation tests of quartz of different particle size were carried out at different impeller velocities below the Cvs. One could expect that increasing agitation should improve recovery due to a better dispersion of particles. However, no such an improvement was evident for coarse particles. A plausible explanation is that the better dispersion is counter balanced by a lower stability of bubble-particle aggregates due to turbulence. In Table 19 spiral feed recovery as a function of impeller speed is presented.

Table 19.
Spiral feed flotation as a function of impeller speed in Agitair cell.
Tall oil 1.2 kg/ton, fuel oil 2.4 kg/ton, frother 30 mg/l. Air 2000 ml/min.

RPM	BPL Recovery %	Grade BPL %
600	58	67
700	75	67
800	59	55
900	35	55

Recovery reaches a maximum at 700 rpm and decreases for higher or lower agitation intensities. At 700 rpm or lower, dispersion of particles was not complete and at 600 rpm or lower, dispersion of air appeared to be insufficient. Additional results are presented in Chapter 3.

As a conclusion we can say that the critical agitation speed for coarse phosphate particles is higher than the agitation speed at which turbulence starts to destabilize the particle-bubble aggregates. Thus, increasing energy input to the cell keep particles from settling but results in lower recoveries. Alternatively if the agitation is reduced recovery might increase, (up to a point), but at the expense of sanding the bottom of the cell. Different impeller design or tank geometry might alleviate this problem, but these tests show that there is an inherent limitation of conventional cells in the treatment of coarse particles, and that avoiding turbulence should be a major concern in coarse particles flotation.

Flotation of Pure Quartz

Quartz flotation tests were carried out in the laboratory column in order to evaluate the effect of variables such as feed and air flowrates and column height. This model system was chosen because it is simpler, more reproducible and easier to work with than the commercial quality phosphate system. A pure quartz sample analyzing 99.9% insolubles and hexadecyl trimethyl ammonium bromide (CTAB) collector were used. Particle size distribution of the quartz is shown in Table 20. As seen there this sample is even coarser than the coarse phosphate feeds used in this study. Tap water at natural pH containing 30 mg/L of aerofroth 65 was circulated through the column at a velocity of 11 cm/min (430 ml/min). Unless otherwise indicated air flowrate was 1430 ml/min and feed flowrate 200 g/min of quartz at 70 % solids.

Table 20. Particle size distribution of coarse quartz.

Mesh (Tyler)	% Retained
10	9.9
14	29.3
20	29.2
28	24.1
35	6.8
48	0.5
-48	0.1

In Figure 3 quartz recovery for three different collector concentrations as a function of column height are presented. In this figure "height" means the distance between the feeding point and the aerator. In previous testing a 55 cm height was standard. It can be seen that by increasing the height to 130 cm, recovery increases about 10-15 %.

It is also clear in figure 3 that collector concentration is extremely important and that total recovery is possible with a short column if the collector dosage is high enough. According to these data a full size column of just 1.5 to 2.0 meters should provide adequate residence time to float a properly hydrophobized mineral. In this respect far more important than the residence time seems to be an adequate collector dosage.

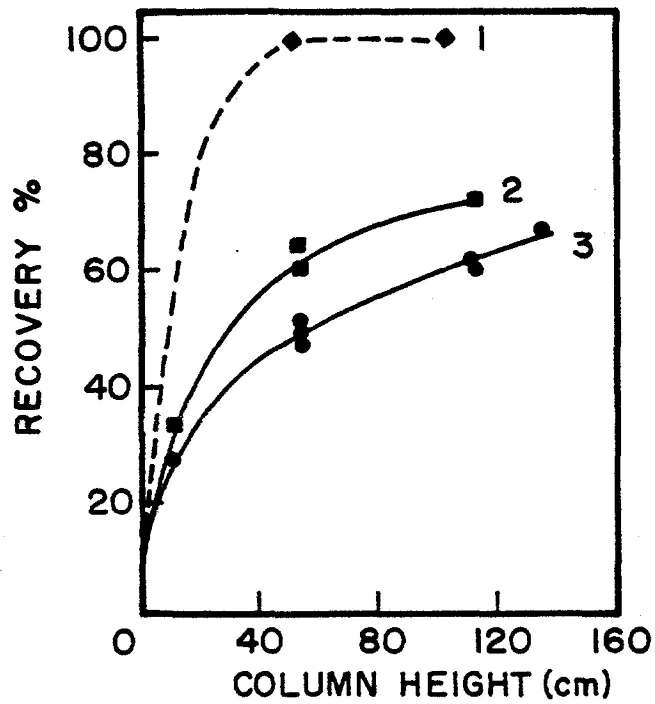

Figure 3. Effect of column height on quartz recovery with CTAB 90 mg/l (1); 50 mg/l (2); 35 mg/l (3).

As shown in Figure 4 air flowrate is also an important variable, higher air flowrates yielded higher recoveries. The maximum air flowrate tested corresponds to air velocities of about 1.3 cm/s (for the 7 cm diameter column this corresponds to 3.0 L/min) well within the air velocities used in conventional column flotation. During phosphate flotation air velocity used was normally only 0.7 cm/s, this led us to believe that increasing the air velocity would increase phosphate recovery. However, as discussed earlier, phosphate flotation actually decreased when air velocities higher than about 0.4 cm/s were used. This points to an important difference between conventional column flotation (where air velocities are as high as 2.0 cm/s) and column flotation of coarse particles.

In Figure 5 the effect of feed rate on quartz recovery is presented. It is seen here that up to a feed rate of 0.65 kg/min there is no effect on recovery. For a column volume of about 3.0 L this corresponds to a capacity of about 10 kg/hour per liter of cell, roughly comparable to the capacity of a conventional cell. Higher feed rates were not easily tested due to limitations in the feeding apparatus. Maximum phosphate feed rate tested was about 0.5 kg/min.

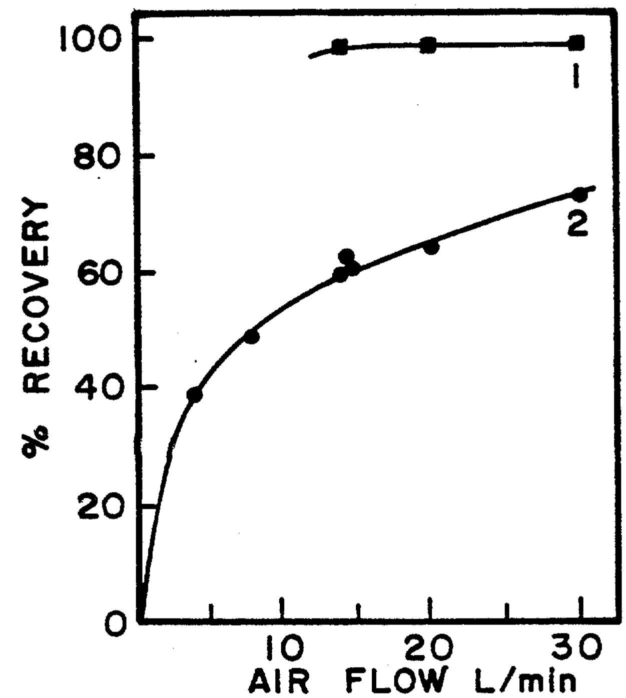

Figure 4. Effect of air flowrate on quartz flotation with CTAb 90 mg/l (1) and 50 mg/l (2).

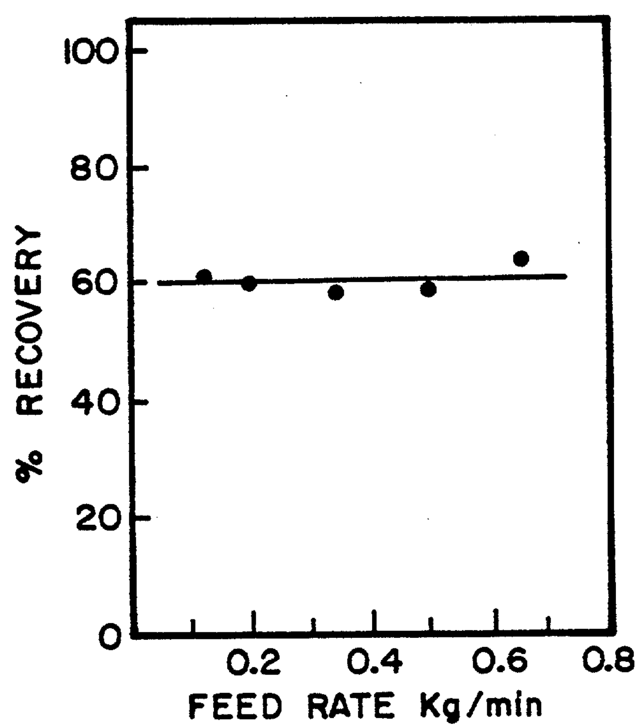

Figure 5. Effect of feed rate on quartz recovery. CTAB 50 mg/l

Effect of Froth Depth.

A series of tests with single size quartz particles were performed in the 7.0 cm column with froth depths of 0, 1.0 and 5.0 cm. Results are presented in figure 6. In the same figure results obtained under similar conditions in a conventional cell are also given. Two main conclusions can be obtained from these data. In the first place it seems clear that thickness of the froth decreases recovery of the larger particles during column flotation. The effect, however, is much less important for the finer particles. It is also evident that the column with a thin froth layer (1 cm) yields much better recoveries than a conventional cell with similar froth depth. This difference is accentuated for the coarser fractions and tends to disappear for the finer particles.

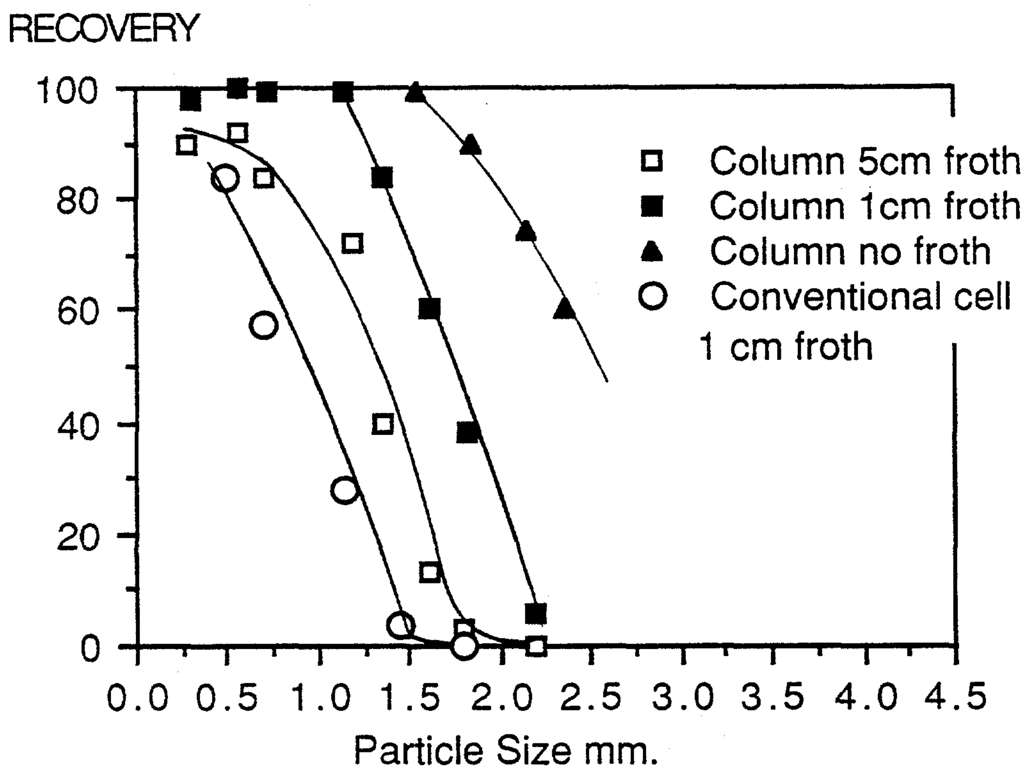

Figure 6. Effect of froth depth on the flotation of pure quartz in conventional cell and in 7.0 cm column. Collector concentration 700 g/ton

Entrainment of fine quartz.

The entrainment of quartz particles by an upwards flow of water was determined in column flotation test performed in the presence of frother but without addition of any collector. Some tests were carried out with single size fractions and others with a sample of a size distribution shown in table 21. One kg wet samples were fed to the column like in a normal flotation experiment. Unless otherwise indicated air flowrate was 1500 ml/min, feeding time was 5 minutes and the feeding tube was located at a depth of 20 cm.

Table 21. Particle size distribution of fine quartz sample.

Mesh (Tyler)	% Retained
28	1.9
35	16.0
48	34.1
65	23.7
150	20.1
200	3.0
-200	1.1

As shown in table 22, water flowrate was the most important factor in controlling entrainment, on the other hand the effect of air flowrate seems minor.(see also Table 24)

Table 22. Effect of water flowrate on quartz entrainment

Water Flowrate (ml/min)	Entrainment %
0	0.2
200	0.2
400	0.8
800	1.4
1100	2.5

The effect of depth of feeding point is shown in table 23. It is evident from the results shown in this table that entrainment can be controlled by adjusting the depth of feeding and that a few centimetres are enough to reduce the entrainment to negligible levels. The entrainment of particles larger than 150 mesh was practically nil.

Table 23. Effect of depth of feeding point on quartz entrainment at a water flowrate of 800 ml/min.

Depth (cm)	Entrainment %
0	3.4
20	1.6
40	0.9
75	0.2

Table 24. Effect of air flowrate on entrainment.

Air flowrate (ml/min)	Entrainment %	
	Water Flowrate (ml/min)	
	0	430
400	0.99 (?)	
800	0.14	0.73
1500	0.11	
2400	0.16	
3300	0.78	1.5

Residence Time Determinations.

The residence time of individual phosphate particles of various sizes was determined in the column operating as in normal flotation except that no collector was added. A large spread of values was evident due to the shape factor, more flat particles had a terminal velocity much lower than more spheric particles. However, the spread tended to decrease for the smaller sizes. The comparison of the average experimental values for each fraction with theoretical calculations assuming spherical shape indicated a relatively large discrepancy for the coarser sizes, but the discrepancy decreases for the smaller sizes. The theoretical value was nearly the same as the experimental value for the smaller fraction tested (35x48 mesh). Since experimental determination with particles smaller than 48 mesh was very difficult, for this type of particles the theoretical values can be considered a good approximation . Average values for different particle sizes are given in Table 25.

Table 25. Residence time estimation for phosphate particles of different sizes.
Air flowrate 1500 ml/min, water flowrate 600 ml/min (0.26 cm/s).

MESH	SIZE (mm)	SETTLING VELOCITY (cm/s) Experimental (2)	Theoretical(3)	RESIDENCE TIME (1) Experimental
+14	1.2	14	25	3.9
14x20	1.0	12	21	4.6
20x28	0.7	9.8	13	5.6
28x35	0.5	7.5	8.5	7.3
35x48	0.36	6.4	6.1	8.6
48x65	0.25	-	4.3	12.8 (theor.)
65x100	0.18	-	2.6	21.0 (theor.)

1) Seconds in flotation zone 55 cm long.
2) Average value of at least 40 measurements.
3) Assumes average size spherical particles and 10% air hold-up, i.e. density of the fluid 0.9 g/cc.

CHAPTER II

PILOT TESTING

COLUMN SCALE UP

The conventional scale up procedure for conventional columns involves the experimental determination of the flotation rate constants, the so called “carrying capacity” limitation, and then an estimation of the axial dispersion factor that reduces the residence time of the mineral particles in the collection zone of the column. More recently a lip discharge limitation has been considered for very large diameter columns. In the case of a frothless column to treat coarse particles any limitation by carrying capacity or lip discharge can be neglected. Also it can be assumed that the flow regime of the particles is plug type so no dispersion factors or short-circuiting problems need to be considered. If this is the case, the residence time of the particles is simply the time that it takes the particles to settle from the feeding point down to the aerator level at the bottom. Residence time is then a direct function of column height, particle size and density.

If we accept these basic points, the scale up procedure is quite simple: Capacity of the column is scaled up as a direct ratio of the column surface. Column length is maintained essentially constant in order to keep a constant residence (i.e. flotation) time. (In practice some allowance for a good radial distribution of air and feed would make it necessary to increase the column length as the surface of the column is scaled up.). Operating variables such as air and water flow rates and sparger surface should also be scaled up as a direct function of column surface. In other words this means to maintain constant air and water velocities and a constant aerator surface/column surface ratio. Accordingly, to scale up our laboratory column 7.0 cm diameter to a 1.0 foot unit we used the following criteria:

Column length (collection zone only): 4 feet. (This would give a residence time of the order of 10 seconds).

Cleaning zone: 1 foot

Aerator surface: At least 50% of the column surface

Air Velocity: 0.6 cm/s

Water Velocity: 0.5 cm/s

Estimated Capacity: at least 0.5, tph (based on a laboratory capacity of about 0.7 tph/sq.foot)

THE PILOT COLUMN

The column body was made of a transparent plexiglass tube (1.0' external diameter) to facilitate visual observation. The conical bottom was made of stainless steel l/4" thick. The body was divided in three sections. Two intermediate sections 4' and 2.5' long respectively and an upper section 1.5' long with a squarely shaped concentrate launder. The different sections were sealed with rubber gaskets and bolted together. Feeding was done by introducing a two inch diameter tube at the centre of the column to a depth of about one feet. The zone of the column above the end of the feeding tube can be considered a "cleaning zone". For most of the testing only one of the intermediate sections was used, thus the column length was just 5.5'. Out of this, about one foot corresponded to the "cleaning zone", and therefore the collection, or active zone, was just 4.5' long. A diagram of the column is shown in Figure 7. As seen there, the conical bottom section has two spigots to add the water necessary to obtain the elutriation effect. It also has a 1 inch valve for regulating the tailings flow. The column is selfsupported by a 3'x3' mobile structure made of square steel tubes.

Instrumentation incorporated to the pilot column was quite modest, but enough for a proper control during operation. Instrumentation included flowmeters to measure the air and water flowrates, manometer to determine air pressure and a manometer connected to the column to estimate air hold up. During testing variables such as feed, air, and water flows were kept constant and tailings flow rate adjusted manually to the minimum necessary to avoid accumulation of tailings in the bottom of the column. This way an upwards flow of water was always prevalent in the column. A commercial column should have automatic control of the tailings flow because fluctuations in the feed rate or feed grade make it necessary to adjust quite often the tailings flow rate in order to keep a steady state. Other variables do not need frequent regulation and they can therefore be set to the desired value manually.

After the pilot test was completed, additional instrumentation was incorporated to automatically control the tailings flow. This is shown in Figure 8 but it has not been tested in an actual continuous operation.

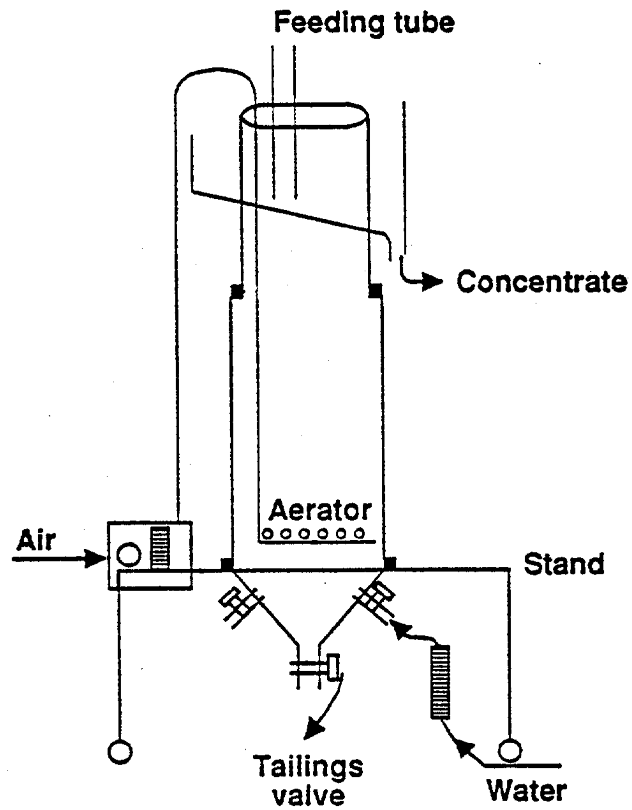

FIGURE 7 Diagram of pilot column

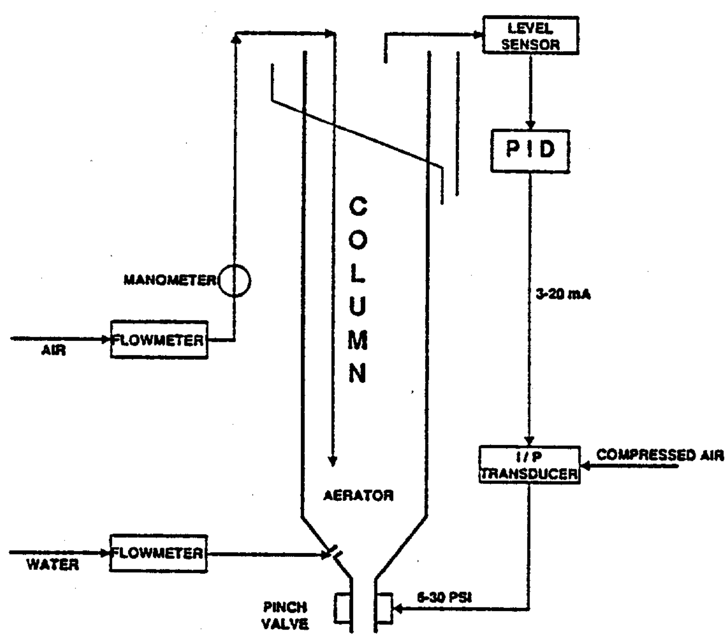

DIAGRAM OF COLUMN WITH CONTROL ELEMENTS

FIGURE 8.

Sparger Design

As shown in Chapter 1 bubble size is one of the over riding factors in column flotation. In order to extend the laboratory results to a larger column, the aerator surface has to be scaled up such that the ratio aerator surface/column surface is at least maintained. In general the larger this ratio the smaller the bubble size obtained for a given air flow rate. Also, the physical configuration of the aerator has to be favourable for an even distribution of air across the column section.

Column type flotation machines are quite simple and consume almost no energy because they do not have any moving mechanical parts. If we do not consider the accessory control equipment, the only sensitive element in a flotation column is the aerator or sparger. Aerators used in flotation columns can be divided in two types:

Type a). Bubbles are obtained by forcing air through a porous media such as: filter cloth, perforated rubber, and other porous materials such as porous plastics, ceramics or metals.

Type b). Injection of pressurized air and water gives origin to fine bubbles by de-compression and shearing forces obtained in static mixers, fine orifices or nozzles.

A common characteristic of all types of spargers is that they are a fixed feature of the column. There is little or no flexibility at all to increase or decrease its number, or the total surface area of the porous media or to change their position inside the column.

Spargers made with porous media such as inexpensive filter cloth were among the first to be used. They are made in different forms and size such as sections of disc filters or perforated iron tubes covered with filter cloth. They are introduced inside the column, close to the bottom of it, through special holes cut on the column wall. Some of them include valves and are designed such that they can be removed and replaced through the column wall. Their total surface area is relatively low and therefore they tend to give relatively large bubble sizes. Also, these aerators tend to leak in the joints which are sewed together and the cloth tends to tear. Rubber aerators also tend to tear in the joints. Porous metals are strong and practically leak proof but they are expensive and they clog easily. Since aerators are introduced through the wall of the column, it is quite difficult to change its number, size or position once the column has been built. For these reasons a new kind of aerator was designed for the pilot column.

This new aerator uses the same type of porous media known before, but it has a number of advantages over the different spargers or aerators used so far in flotation columns. It can be made with porous tubes (plastics or steel) or tubes covered with filter cloth or any other porous media than can be interconnected to make a framework of porous elements. Also, the framework of porous media can be assembled without any internal tubing such as in the case that filter cloth (or rubber) sleeves are used. These have the advantages of requiring no gluing and being flexible, which facilitates the installation (or removal) in case there are internal obstacles in the column such as baffles, sensors, feeding tubes etc.

For the particular application in phosphate flotation we chose filter cloth as porous media mainly for its low price. The main feature of this new aerator is that the porous media assembly is introduced through the top of the column. This allows a great deal of flexibility regarding replacement, modifications and maintenance of the sparger. These operations could all be made while the column is operating. One single aerator could have more than one framework of porous elements connected to a single air tube at different heights. In this way the total surface of porous media can be very large and therefore smaller bubbles and/or higher flow rates can be obtained.

The aerator tested in the one foot column (see figure 10) was made with 1/2" cpvc pipe (1) with 1/8" perforations (2). The filter cloth was fixed to the perforated cpvc tubes with a cyanoacrylate type glue so no sewing or clamps were necessary. No leaks were observed and the glued joints of the sparger were able to stand the maximum pressure tested (50 psi). Working pressure of the sparger would be about 12 to 20 psi depending on column height. Dimension of the aerator was just a few millimetres smaller that the column, so once inside the column it had little room to move sideways. This way the aerator was held in place by simply holding the air feeding tube (3) (also made of 1/2" cpvc tube) at the top of the column. The air feeding tube (3) was divided in two sections which were connected with standard male/female fittings (4) of the type used for domestic tap water lines. In case of a longer column the tube could have several sections so it can be more easily handled when being introduced or removed from the column. All connections (4) were tight just by hand and were leak proof even at 50 psi. In a matter of seconds, using bare hands the aerator can be installed, replaced, additional aeration elements can be added or height inside the column can be changed.

Evidently the framework shown in Figure 9 is just an example of many arrangements possible. Another couple of examples appropriate for a square column and a circular column are presented in Figure 10a and 10b respectively. The sparger fits loosely inside the column walls (5) so it can be easily moved in the vertical direction but it is not free to move sideways. The sparger framework is connected to the air feeding tube (not shown in this Figure) by means of a male/female connection (4).

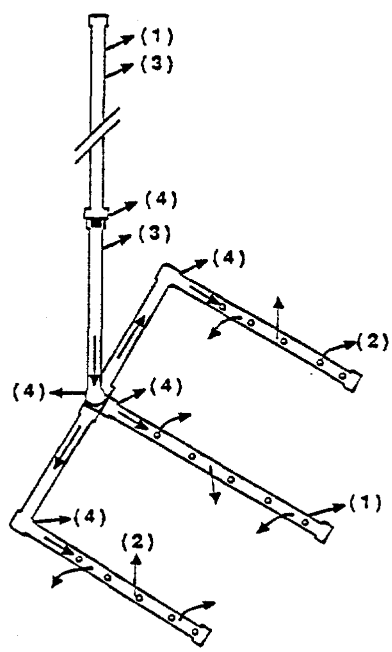

FIGURE 9. Diagram of sparger

Among the advantages of this new sparger we have:

a) Flexibility: It is easy to replace, to add additional surface, and to locate at different heights inside the column. These last two aspects are particularly interesting in kinetics and development studies and also for optimization.

b) Low cost: Materials used are quite inexpensive. Just one manometer and a single flowmeter are enough to control a large size aerator. Column construction costs can be reduced (no holes are cut in the column wall and no aerator-admitting tubes or valves are required).

c) Performance: Finer bubbles can be produced because the total surface area of the aerator can be increased practically at will by adding additional sparging area. Perfect distribution of bubbles across the surface of the column. This is an important consideration in large diameter and/or short size columns. Bubble generation does not require the introduction of water so level control is simplified

d) Avoids flooding of air line in case of air pressure failure. To re start it suffices to re-connect to the air line and slowly increase the air pressure. Can be replaced in a matter of seconds without spillage and without shutting down the column.

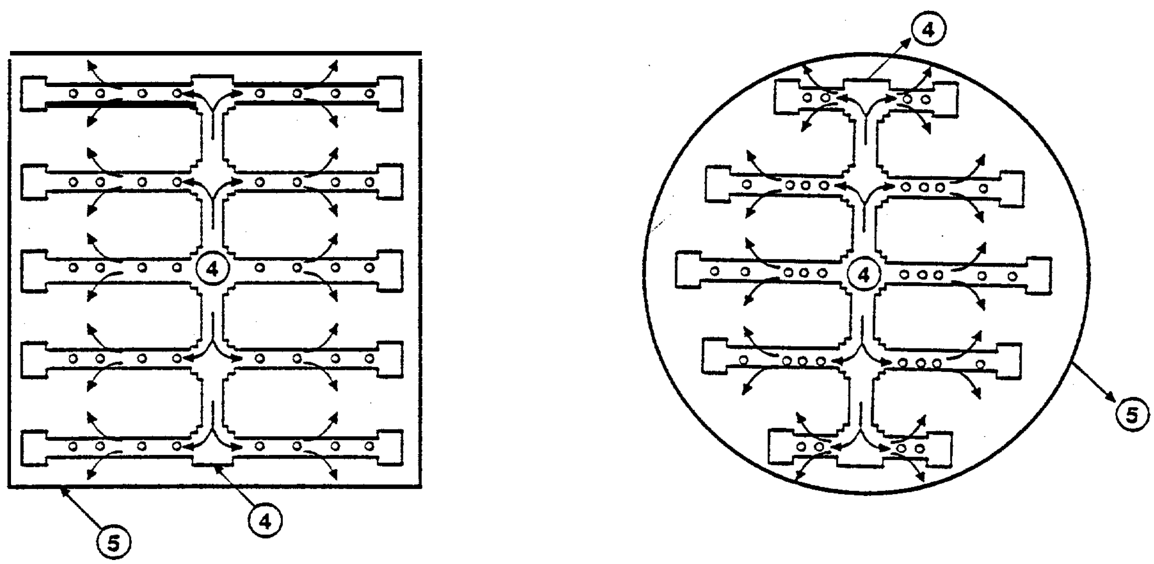

FIGURE 10. Diagram of sparger for square and round column.

Description of the Pilot Test

The column was installed in the spirals section of IMC’s Noralyn plant and fed by gravity with spiral feed taken directly from one of the feed distributors by means of a 3' diameter pipe. No reagents at all were added to the feed and this was sent “as is” to the column. We did not have any control on the amount of reagents or the pulp density or the particle size distribution of the feed. For this reason it can be argued that this was not necessarily a favourable feed for column flotation. For example the pulp density of about 30% was way too low for the best performance of the column. Too much water in the feed means that little additional water can be added at the bottom of the column to achieve the net upwards flow of water necessary for elutriation of bubble-particle agglomerates. Instead, feed water short-circuits directly to the concentrate contributing to entrainment of fine sand. In spite of these less than ideal characteristics of the feed, the pilot test can be characterized as a remarkable success. A total of 17 tests were performed in a period of 5 operating days with feed rates of 0.8 to 1.4 tph. According to information provided by IMC, collector dosage added to the spiral feed during testing was 3.7 and 4.0 lbs per tonne (of concentrate) of fuel oil and soap respectively. The only reagent added to the column was a small amount of frother in the water introduced at the bottom of the column.

Each test lasted for at least a couple of hours in which operating variables were kept constant at a desired value and then samples of the feed, tailings and concentrate were taken for analysis. All samples were analyzed for BPL and insolubles at IMC’s laboratory. Additional chemical assays and particle size distributions were determined at Laval.

RESULTS

The spirals section at Noralyn does not run continuously and frequent interruptions of feeding were experienced. Also some drastic changes in the feed characteristics occurred during testing. For example the first couple of days the feed was high grade (42% BPL) and coarsely sized. After that, feed was lower grade (36% BPL) and finer. Particle size distributions of both types of feed are presented in Tables 26 and 27 . The best results were obtained with the higher grade feed.

Table 26 Particle size distribution of phosphate feed (high grade).

Mesh (Tyler)	% Retained	Cumulative
20	42.1	42.1
28	29.9	72.0
35	23.2	95.2
48	4.1	99.3
65	0.5	99.8
-65	0.2	100.0

Table 27 Particle size distribution of phosphate feed (low grade).

Mesh (Tyler)	% Retained	Cumulative
20	32.4	32.4
28	33.3	65.7
35	26.2	91.9
48	6.1	98.0
65	1.5	99.5
-65	0.4	99.9

For example in Table 28 results of test 2c are presented, phosphate recovery was an outstanding 98.7% with only 8.7% insols in the concentrate. Particle size distributions of tailings and concentrate for this test are shown in Tables 29 and 30 . The negligible amount of phosphate that did not float, was found in the extremely coarse fraction. A sample of the tailings of this test were taken to the laboratory and floated, no additional phosphate was recovered. This shows that recovery of the phosphate values obtained in the column was practically total.

Table 28 Metallurgical results for test # 2C

	BPL %	INSOL. %	BPL RECOVERY (%)
FEED	42.29	43.33	
CONC.	68.82	8.74	98.74
TAILS	1.36	96.6	

Table 29 Particle size distribution of concentrate from flotation 2C.

Mesh Tyler	% Retained	Cumulative	Assay BPL %
20	56.0	56.0	70.8
28	27.7	83.7	70.2
35	12.2	95.9	69.5
48	2.7	98.6	68.6
65	0.9	99.5	15.2
-65	0.5	100.0	4.8

Table 30 Particle size distribution of tailings from flotation 2C.

Mesh Tyler	% Retained	Cumulative	Assay BPL %
20	11.1	11.1	3.0
28	32.9	44.0	1.2
35	44.7	88.7	1.0
48	9.9	98.6	0.6
65	1.3	99.9	0.4
-65	0.1	100.0	0.3

With the lower grade feed, recoveries were also very good but concentrate grades were lower. See for example Table 31. Most of the sand found in the concentrate was present in the fine fractions. As seen in Table 32 by eliminating the -48 mesh fraction from the concentrate, grade increases from a content of 10.7% insols to just 8.3% insols. BPL losses in the minus 48 mesh concentrate are practically negligible. Therefore a simple solution to the entrainment would be to separate (and recycle) the -48 mesh concentrate by screening. (This could be done quite efficiently by incorporating a DSM screen in the concentrate launder of the column).

The sand reporting to the concentrate was probably carried by the large volume of feed water. If the pulp density of the feed is increased drastically say, to 70% solids (i.e. directly from the conditioners), the amount of feed water reporting to the concentrate would be minimal and the sand entrainment should be reduced accordingly. Therefore, the feed for column flotation should be as thick as possible, for the water contained in the feed serves no useful purpose, it is the water introduced at the bottom of the column that helps to levitate the heavy particles.

Table 31 Metallurgical results for test # 3

	BPL %	INSOL. %	BPL RECOVERY (%)
FEED	36.25	50.5	
CONC	64.16	13.69	98.28
TAILS	1.40	97.01	

Table 32 Assay of column concentrate by size fraction. (Test # 14, low grade feed).

Concentrate	Weight Distribution	Insols %	Distribution BPL %	Recovery %
Over all	100	10.7	100	93.1
+ 48 mesh	96.8	8.3	99.3	92.5
- 48 mesh	3.2	82.0	0.7	0.6*

* recovery calculated over the total feed.

Column versus spirals.

The column outperformed the spirals consistently, with all feeds tested. As shown in Table 33 column recoveries were about 30% higher than spirals recoveries, concentrate grades were similar. Spiral and scavenger flotation data presented in this table were provided by IMC and correspond to average plant results during the same period of time that the column was being tested. If the over all recovery of spirals plus scavenger flotation is considered, column flotation was still higher in most situations. At worst column recovery was equal to the recovery obtained in the spirals plus scavenger circuit, in spite of the fact that, as pointed out above, feed characteristics were far from the optimum required for column flotation.

In addition to the low pulp density, the proportion oil/soap was probably not the best one for the column. This is a deduction from the fact that bubble size in the presence of feed appeared to increase considerably with respect to the bubble size obtained in pure water.

This could be attributed to the deleterious effect of excessive oil on the frothing characteristics of the pulp.

Table 33 Comparison of column flotation with conventional spiral circuit.

	Feed BPL %	Recovery BPL %	Insols %	BPL Recovery Spiral + Scavenger flotation
Column	43.3	98.7	8.7	
spiral	43.0	65.2	8.7	93.5
Column	33.5	93.1	10.7	-
Spiral	37.2	62.7	10.8	92.9
Column	36.3	98.3	13.7 *	-

* If the -48 mesh concentrate is discarded insolubles are reduced to 8.5% while BPL recovery remains at 96.1%.

Effect of Frother

One of the first variables studied during the pilot testing was frother concentration. Results are summarized in Table 34 which clearly shows that a small amount of frother is needed to assure a proper air dispersion. When no frother was added BPL recovery was just 78% (still higher than spiral recovery). With just 60 g/ton of frother added to the water introduced at the bottom of the column, recovery increased to 94.3%. The frother used was Aerofroth 65 (polyglycol type frother from Cyanamid) and a dosage of 30 g/ton was kept constant for all subsequent testing. A frother of the same type (polyglycol) provided by IMC was tried in some later test with results similar to those obtained with the aerofroth 65.

These results show once more that one of the most sensitive factors in column flotation is bubble size. In addition to the effect of the frother, bubble size depends on factors such as efficiency of the aerator, air flow rate and the presence of oils.

Table 34 Effect of frother on phosphate recovery and concentrate grade.

Frother (gr/ton)	Recovery BPL %	Grade BPL %	Grade Insols %
0	78.2	68.5	8.1
30	91.7	69.0	8.7
60	94.3	68.9	8.7

Effect of Air Flow Rate

Air flow rate is probably the most important operating variable. The flotation time in a column just 4 feet long is so short that a maximum probability of bubble-particle collision has to be achieved. Therefore, in its fall to the bottom of the column the particles should find as many bubbles as possible. This means that the air hold up (i.e. the proportion of total column volume occupied by the bubbles) should be as high as possible. The air hold up is a function of the air flow and the bubble size: the larger the air flow and the smaller the bubbles the higher the air hold up. In table 35 data of air hold up measured in the pilot column in different conditions are presented. Hold ups obtained in the presence of frother are very high, which demonstrates the efficiency of the sparger. However these data corresponds to hold up values in the absence of mineral particles. When feed was added to the column air hold ups decreased considerably (see Table 36). This is probably at least partially due to the effect of reagents in the feed on the bubble size. It is likely that the oil present in the feed increases the bubble size. If this is the case, some optimum proportion of soap/oil should be used for column flotation such that hydrophobicity of the phosphate is achieved without an undue effect on bubble size.

From hold up considerations alone, it could be argued that very high air flow rates should be used for column flotation. However there are at least three factors that limit the maximum air flow rate that can be used: turbulence level and bubble size increase with increasing air flow rates, and density of the fluid (and therefore its levitation capacity) decreases.

The effect of air flow rate was studied in several tests. For example in Figure 11 we see recoveries and concentrate grade as a function of air flow. (all flow rate data are corrected to standard conditions, in actual testing pressure varied from 9 to 16 psi depending on column depth, characteristics of the sparger and the air flow). A wide optimum range of 20 to 40 liters per minute (lpm) corresponding to air velocities of about 0.5 to 1.0 cm/s was observed. This optimum range of air flows is in agreement with laboratory results and points to a definitive difference with conventional column flotation that requires air velocities of 2.0 to 3.0 cm/s. With air velocities below 0.5 cm/s a drastic loss of recovery was observed, after all no air means no recovery. Air flows higher than about 1.0 cm/s yield slightly lower recoveries, this is probably due to excessive turbulence and/or to an increase in the bubble size. To scale up these pilot test results to a full size column the factor 0.8 cm/s for air flow rate could be used. Concentrate grades were not affected to any large extent by the air flow rate.

Table 35 Air hold up % as a function of frother concentration and air flow rate.

Air Lpm	hold up % No frother	hold up % 20 ppm frother
5	1.2	7.4
10	2.1	3.6
15	3.3	5.5
21	4.2	7.0
26	5.2	8.5
32	5.8	9.7
28	6.7	11.2
45	7.9	13.0
51	8.2	14.5
57	8.5	16.4

Table 36 Effect of phosphate feed on air hold up.

Hold up % In water	Hold up % In presence of feed
7.4	1.7
12.4	3.3
16.1	5.4
17.5	5.8
19.0	7.8

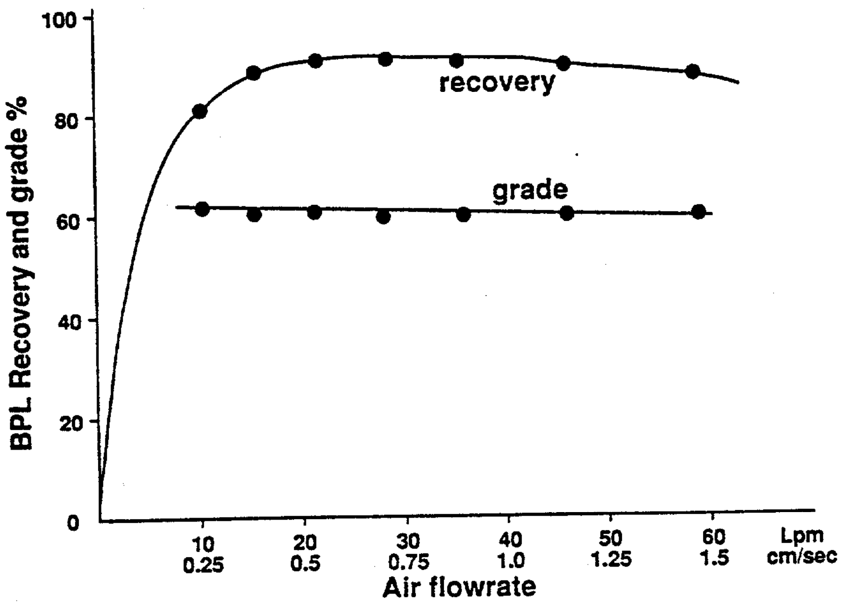

FIGURE 11. Effect of air flow rate of phosphate recovery and grade.

Effect of the Sparger.

The advantage of having a sparger that can distribute the air all across the column can not be over emphasized. Also the flexibility of being able to control the air flow independently from the water flow is a very desirable feature. The sparger used proved to be quite efficient. In laboratory testing the sparger did not loose any effectivity even after two weeks of continuous bubbling in a phosphate pulp. During testing, performance of the sparger was also quite steady. A small loss of porosity was observed when the sparger was left immersed (without bubbling) for 48 hours in plant water, it appears that some algae grew in the cloth. It is probable that the same does not occur when air goes through the aerator because no problems were observed during operation. Anyways porosity of the partially clogged aerator was restored by simply washing with a jet of water.

During the pilot test the effect of the aerator surface was studied. Results of this study are presented in Tables 37 and 38. In the first table air hold ups obtained with three different aerator

surfaces as a function of the air flow rate are presented. Differences in the air hold up values of tables 37 and 38 are explained by the fact that data in table 37 were obtained in water while those of table 38 were measured with pulp. It is seen in table 37 that by increasing the aerator surface larger hold ups can be obtained. This means that the bubble size is smaller when the aerator surface increases, and therefore better recoveries should be obtained. In table 38 it is clearly demonstrated that higher recoveries are obtained with larger aerator surfaces.
As a conclusion we can say that to scale up a column, surface of the aerator should increase proportionally to the surface of the column, maintaining a ratio column/aerator of at least 0.5 but preferable closer to 1.0.

Table 37 Air hold up (%) as a function of aerator surface and air flowrate.

Air	Aerator surface cm^2		
Lpm	107	322	534
10	2.9	4.1	4.5
20	4.9	8.6	8.7
30	6.6	12.4	13.6
40	7.4	16.1	17.4
50	9.1	19.0	21.5

Table 38 Effect of aerator on phosphate recovery and concentrate grade.

Aerator (cm^2)	Surface ratio: column/aerator	Air hold up %	Recovery BPL %	Grade BPL %
107	0.2	1.7	49	70
322	0.5	5.4	92	68
534	0.8	5.8	93	69

Effect of Water Flow Rate

The effect of water flow rate is summarized in Table 39 and Figure 12 . Although, data in this table are obtained from different experiences in which the other operational variables are not necessarily the same, a clear tendency towards higher recoveries when high water flow rates were used is evident. In these data water flow rates refers to the amount of water that reports to the concentrate. The majority of this water comes from the feed and only a small proportion corresponds at the water introduced to the bottom of the column. This is due to the fact that the feed was quite diluted, With a more dense feed material, a much large proportion of water could be added to the bottom of the cell for the same overall water flow shown in Figure 12 . In these conditions the entrainment factor would be the same as in Figure 12 but recoveries could increase considerably. As a probable operating value we can estimate a total flow of water to the concentrate of the order of about 1.5 cm/s which results in some 23 gpm per square foot of column. Depending on the content of fine sand this value could be increased or decreased. The higher proportion possible of this water should be coming from the water introduced at the bottom of the column.

TABLE 39. Effect of water flow rate on recovery and grade.

Exp #	Water flowrate		BPL Recovery %	Concentrate Grade	
	cm/s	Lpm		Insols %	BPL %
9A	0.2	8	67	6.3	70.2
10A	0.27	11	89	7.9	68.7
6	0.4	16	78	7.3	69.0
7A	0.4	16	74	7.1	69.2
9B	0.7	28	92	8.3	69.6
5	1.4	56	91	9.8	67.9
7B	1.6	64	90	9.3	68.4
10B	1.7	68	93	12.8	65.0

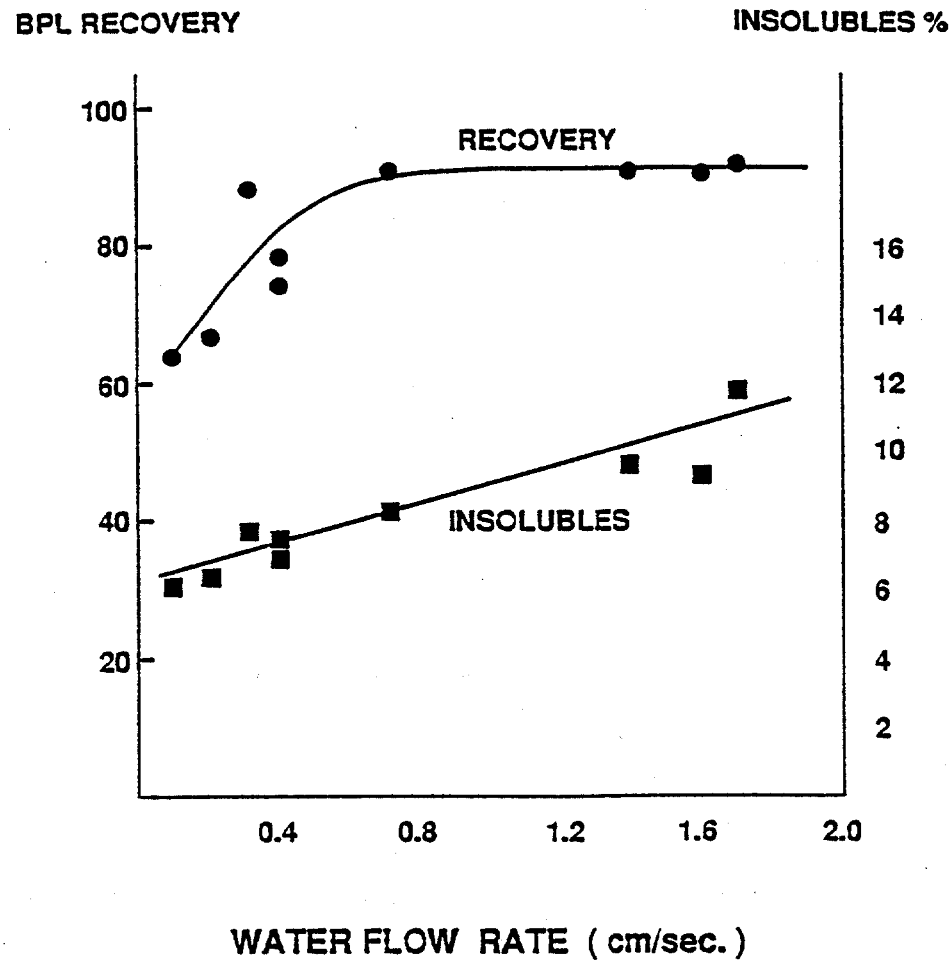

FIGURE 12. Effect of water flow rate on phosphate recovery and grade.

Effect of Flotation Time

The effect of column length was evaluated by changing the height of the aerator inside the column. Accordingly the distance between the feeding point and the aerator i.e. the “effective column length”, also changed. In Figure 13 recoveries obtained for different “column lengths” are represented. It is seen in this figure that recovery increases rapidly as the collection zone of the column increases up to about 60 cm when nearly 90% BPL recovery is obtained.
This means that the flotation rate is extremely fast. Thus most of the floatable values are recovered in a few seconds. Very little of the phosphate contained in the feed has the opportunity to reach beyond the first couple of feet inside the column. For this reason the pulp density inside the column is not uniform, it is more dense at the top and more diluted at the bottom.

The flotation kinetics obtained in the pilot test is in agreement with that obtained in the laboratory study. The probable reason for the fast flotation in the column is the combination of favourable conditions: no froth, low turbulence and a high concentration of small size bubbles. This fast flotation kinetics means that a full size column does not need an active zone longer than some 5 feet. Allowing one to two feet of cleaning zone, plus a couple of extra feet to get a good distribution of the feed and to dissipate the turbulence caused by the entrance of feed, total height for a full size column should be about 10 feet.

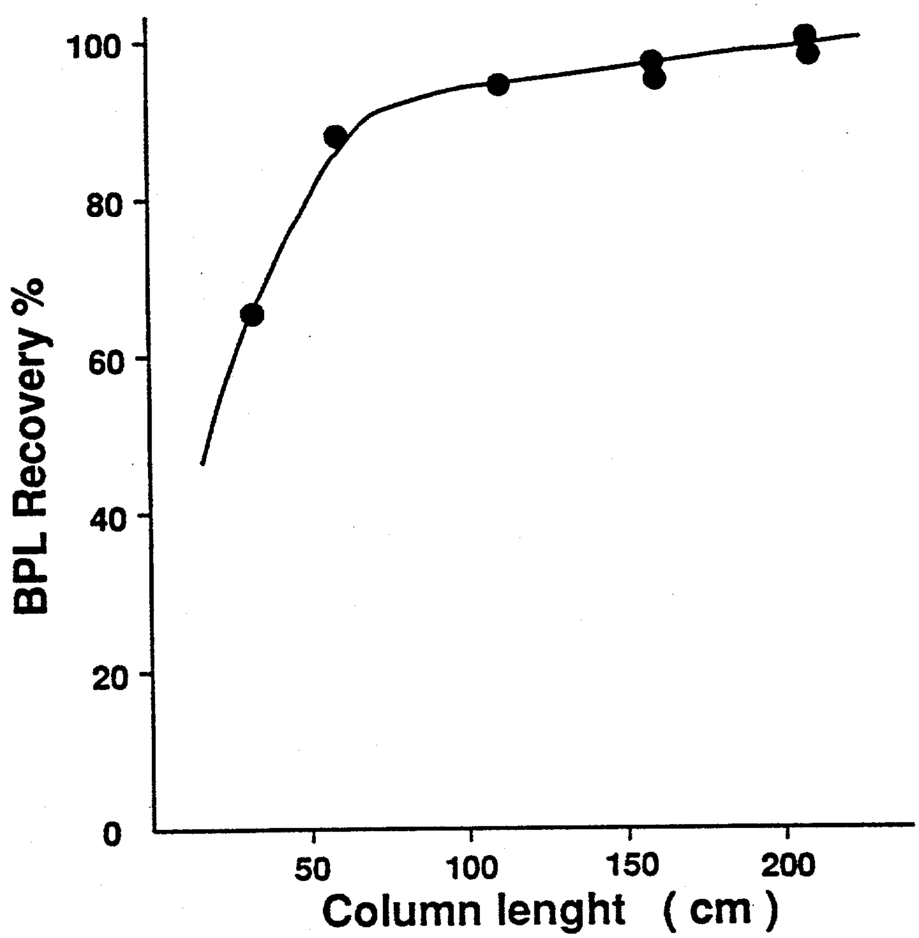

FIGURE 13. Effect of column length on phosphate recovery.

Effect of Feed Rate.

Most of the column tests were performed with a feed rate of 0.7 to 0.8 tph (metrics tons of dry solids per hour). As shown in table 40 when feed rate increased beyond 0.8 tph recovery decreased. Unfortunately we had little control over the feed characteristics so no other feed conditions were tested. The limiting factor in these tests was the larger amount of water introduced to the column with the larger feed rate. This created turbulence in the feeding region, and increased the amount of water reporting to the concentrate which in turn limited the amount of water that could be introduced at the bottom. Therefore, it is our feeling that by optimizing the pulp density of the feed and the ratio soap/oil, and by slightly increasing the column length and adjusting the operating conditions in the column, recoveries for a feed rate of at least 1.0 tph should be similar to those achieved with 0.8 tph. If this proves correct a likely scale up factor for column capacity would be 1.4 tph/square feet. (Note that this figure is twice that estimated from the laboratory study).

Table 40 Effect of feed rate on phosphate recovery and concentrate grade.

Feed rate tph	Recovery BPL %	Concentrate Grade BPL %
0.7	93	66
0.8	96	68
1.0	86	69
1.4	83	70

FINAL REMARKS

It is clear that some of the features of the column tested here are shared by the Flotaire cell and we are not surprised to hear of some definitive successes of this cell in the treatment of coarse phosphate. From our experience we can predict that the Flotaire cell should give a best performance when a negative bias is prevalent such that no froth at all is formed at the top, and when an air flow rate of 0.6 to 0.8 cm/s is used (assuming that small bubbles can be obtained). We think, however, that our design is more flexible than the Flotaire cell in the sense that allows a better, and independent, control of air and water flow rates. Thus a better control of bubble size and air hold up can be achieved. Additional advantages of our column are a smaller size, better distribution of air, less consumption of water and total absence of turbulent zones.

Technology of this column is so simple that any phosphate company could build, in house, a full size fully instrumented column for less than the cost of the energy that an equivalent mechanical cell would consume in a year. Operation seems to be quite simple and there is no consumption of energy (except for the small energy required to get compressed air at just 15-25 psi.). The only sensitive factors being those related to bubble size and air hold up. As far as control is concerned, the only variable that needs automatic control would be the tailings flowrate. Alternatively, if a low density tailings flow is accepted, manual setting of the tailings valve would suffice. But a low solids discharge has the inconvenience of higher consumption of water and the creation of turbulent zones in the column.

Results of the pilot test suggest that a column to treat 50 tph of coarse phosphate would be just 6x6 feet of surface by no more than 10 feet height. Other characteristics of such a column are presented in table 41. If this scale up is correct it means that just two columns could do the job of a circuit having about 100 spirals plus a conventional scavenger flotation stage.

Additional testing performed with potash ores with this column suggest that the upper limit of floatability of coarse particles can be extended beyond 16 mesh. Considering the density differences between potash and phosphate minerals, the potash test indicates that recovery of phosphate particles up to 2.0 mm would be possible in this column. This would open an interesting perspective to up grade low quality pebble by column flotation.

In summary this project has arrived at an optimistic conclusion. I believe that we have demonstrated the potential advantages of column type cells in the treatment of coarse particles. These results can be extrapolated to suggest that column cells should outperform mechanical cells in the treatment of soft ores such as the dolomite rich ores typical of the southern extension and that feeds coarser than presently treated could be concentrated by column flotation.

Table 41 Scale up of pilot test results to 50 tph capacity.

Column surface	3.35 m^2	(6 x 6 feet)
Column height	3.0 m^2	(10 feet)
Aerator surface	2 m^2	(21 square feet)
Air flowrate	1.5 m^3/min (50 cfm)	
Water flowrate	1.2 m^3/min (300 gpm)	
Recovery	+95% BPL	
Concentrate Grade	7-10% Insolubles	

CHAPTER 3
REPORT ON WORK DEVELOPED DURING THE FIRST YEAR

This chapter includes experimental results and discussion of the test work developed during the project's first year that was not included in Chapter 1. Section 1 contains results and discussion of some exploratory tests performed in a small laboratory column just 2.5 cm diameter. Given the encouraging results obtained at this stage a larger (7 cm diameter) laboratory column was built. The most relevant results obtained in this column were already discussed in Chapter 1. The rest of the experimental results obtained in the 7 cm laboratory column are presented in the second section of Chapter 3. The next section contains results of theoretical studies that include residence time determinations of bubbles and particles as well as air hold up measurements.

Materials and methods were described in Chapter 1. Phosphate products from the flotation tests were dissolved in hot concentrated aqua regia during 30 min. After dissolution, insolubles were filtered, dried and weighed directly in fritted funnels. Phosphate was analyzed in the acid liquor by a colorimetric method involving the formation of a coloured molybdo-vanadate phosphate complex. However, for routine analysis only insolubles were determined and BPL content was calculated from the insolubles assay. This procedure was calibrated and checked with analysis performed in analytical laboratories of Florida phosphate plants. Differences in BPL assays are less than 0.8 % BPL which is precise enough for most of the flotation testing. A key factor to achieve this precision level was the use of relatively large samples during analysis. Flotation products were sampled in a riffle to obtain 20-30 grams of material for analysis. This relatively large amount of material decreases the sampling error of coarse particles and assures very good reproducibility of assays.

EXPLORATORY FLOTATION TESTS.

Conventional Cell

A number of tests were performed with spiral and coarse flotation feeds in both an Agitair and a Denver laboratory cell. Samples of 1 Kg of ore were conditioned in the cell at 60 % solids during one minute with fatty acid (Acintol AF3) and kerosene as extender. Using this procedure no flotation of phosphate was observed even at the highest collector doses added (4 Kg/ton). Results are presented in the appendix t the end of the Chapter. It was suspected that this low floatability was related to the strong agitation used during conditioning (needed to fully suspend the solids). Apparently the strong agitation abraded the mineral surface. Therefore the conditioning procedure was modified, and instead, the ore was tumbled for three minutes in a plexiglass container at 70% solids. With this method phosphate recovery increased markedly. The best results were obtained with the Agitair cell. By using 4 Kg/ton collector, BPL recovery from the spiral feed was 53% with an insols content of 16%. With the coarse flotation feed maximum recovery was 74%, but insols content of the concentrate was 39%.

Column Cell

Exploratory testing was carried out in a small column 2.8 cm diameter and 50 cm height. The column was feed continuously at a rate of 50 g/min with a 60% solids pulp from a paddle stirred tank located on top of the column. Before feeding the ore was conditioned as for conventional flotation. Very good recovery was observed during the fast 2-3 minutes of flotation, but an increasing deterioration of floatability was evident afterwards. It was suspected that this behaviour was due to abrasion of the ore in the feeding tank. Subsequent experiments were performed without stirring during feeding. In this case extremely encouraging results were obtained. Nearly 100% phosphate recovery was obtained with both feeds. Selectivity was higher than in a conventional cell. In Table 42 best results obtained in similar conditions in conventional cells and in the column are compared.

Table 42. Flotation of spiral and coarse feeds in conventional cells (Agitair and Denver cell) and in 2.8 cm column.

Reagents 1.5 kg/ton kerosene and fatty acid for coarse flotation feed and 2.0 kg/ton for spiral feed. Frother 10mg/l.

	Spiral Feed			Coarse Flotation Feed		
	Recovery %	Grade BPL %	Ins. %	Recovery %	Grade BPL %	Ins. %
Column	99	65	13	98	62	17
Agitair Cell	53	62	17	74	45	39
Denver Cell	49	63	15	72	47	36

As seen in the results shown in Table 42 performance of the column is clearly superior to a conventional cell. Particularly, recovery of the plus 20 mesh (0.83 mm.) fraction presented no problems in the column while the same size fraction did not float in the mechanical cells (See Table 43).

Table 43. Particle size distribution of spiral feed concentrates.

Mesh	% Retained Column	Conventional Cell
20	20.1	4.8
28	38.7	34.7
35	25.8	28.8
48	9.7	22.5
- 48	5.7	9.2

Also, concentrate grade obtained by column flotation was much higher than in the conventional cells, in spite of the fact that the relatively high upwards flow of water used in column tests dragged most of the fine sand contained in the phosphate feed. However, this fine sand entrained in the concentrate was easily eliminated by merely screening the minus 48 mesh fraction of the concentrate. In this case grade of the spiral feed concentrate increased to 69 % BPL (8% insols) with a negligible loss of phosphate (See Table 44). This indicates that the column has the potential to produce a final high grade concentrate in a single stage flotation, without deoiling or cationic flotation.

Table 44.
Analysis of column flotation concentrate sized at 48 mesh.

	+48 mesh	-48 mesh	Total
BPL %	68.5	25.5	65.1
Insols%	8.8	66.1	13.0
Recovery %	95.0	4.0	99.0

In the column tests an upwards flow of water was kept constant at 600 cc/min (1.6 cm/s) and the air flow was 300 cc/min (0.8 cm/s). Water from the concentrate was recycled to the column and for each test that lasted about 10 minutes (and in which 500 grams of ore were floated) only 2 liters of water were used.

The most interesting observation during column testing was that when the water flow was shut off and a froth bed of about one inch was allowed to form on the top of the column , phosphate recovery decreased sharply . In this case particles attached to air bubbles raised to the water level but were recycled back to the middle of the column because they were unable to climb through the froth layer up to the rim of the cell and exit the column with the concentrate. Since feeding was continuous, this originated an accumulation of air solid agglomerates near the froth liquid interface that eventually collapsed and ended up in the tailings fraction. Although in this test the pulp level was not well controlled results clearly indicate a lower recovery when a froth layer is formed on top of the column.

Table 45. Recovery of spiral feed in column with and without a water flow .

Water flow	Recovery %	BPL %
0	25.8	68.7
600	98.5	65

Another interesting observation was that flotation kinetics was very fast and collection of mineral particles by raising bubbles took place in the top few centimetres of the column. Residence time of the coarsest particles in the column was only a couple of seconds, nevertheless, very high recoveries were possible. This is quite advantageous for scale up purposes and it means that a full size column need not to be longer than a few feet to give adequate recovery.

LABORATORY FLOTATION TESTS.

In view of the encouraging results obtained with the small column a larger laboratory unit (7 cm diameter) was built. Design considerations and experimental procedures were described in Chapter 1. Also a diagram of the column was presented in Figure 1.(Chapter 1)

Some of the earlier flotation tests were conducted after conditioning the ore for three minutes in a conditioner without internal lifters. These tests showed a rather large experimental error that was attributed to imperfect conditioning. Both the tall oil and the fuel oil were viscous and sticky and therefore difficult to disperse. Results of these tests are presented in the appendix at the end of this Chapter. The procedure adopted next: 8 minutes tumbling in a container with lifters provided a more reliable conditioning with the flotation reagents.

Results

Most of the flotation results obtained with the 7 cm diameter laboratory column were already presented in Chapter 1.
Optimum collector dosage was found to be between 3.0 and 4.0 kg/ton. Very low recoveries were observed for total collector dosage below 3.0 kg/t, on the other hand, at dosages of about 3.6 kg/t recovery was better than 95%. Some typical results including mass balance calculations were shown in tables 3-6 and Figure 2 in Chapter 1. Additional results are presented in the appendix.

These results are quite encouraging and confirmed the preliminary results obtained in the smaller column. Recoveries obtained in the column were always higher than recoveries in the conventional cell. Conditioning procedure, collectors, frother, pH and air flow were the same for both the column and the mechanical cell. Agitation speed in the mechanical cell was the minimum to avoid sedimentation of particles. Tests in a Denver laboratory cell gave slightly lower results than those obtained in the Agitair (See appendix).

Phosphate flotation in the column was extremely fast, about one order of magnitude faster than in a conventional cell. In the conventional cell flotation time was about 60 to 90 seconds, while in the column some ten seconds were enough to achieve nearly 100% recovery.

In addition to the effect of collector, recovery and grade obtained during column flotation depended to a lesser extent on factors such as water and air flowrates, frother concentration, bubble size, and depth of the feeding tube (See Chapter 1 for details). The addition of water was essential to avoid a froth layer on the top of the column. In order to float coarse particles it is necessary to keep a negative bias inside the column (i.e. a net upwards flow of water) to transfer the floating particles to the concentrate without the formation of a froth bed.

Some tests were performed to explore the possibility of recovering the phosphate values by reverse cationic flotation. Exploratory testing in conventional cells and in the small diameter column were not encouraging. Early tests in which samples were conditioned by agitating directly in the cell failed to reduce the insols content when up to 2.0 kg/ton collector (Azamine plus kerosene) were used. Not much improvement was observed when the conditioning procedure was changed to a mild tumbling during 3 minutes. Results in both conventional cells and in the column indicated low recoveries when high amounts of collector were used, or low grades when small concentration of collector were added.

Further testing of the cationic route using azamine and diesel fuel in the 7 cm column gave rather disappointing results, no major improvement over the exploratory tests was observed. This amine type collector was not very selective . If collector dosage is increased in order to float more quartz, fine phosphate starts to float and is lost in the froth product, but coarse quartz remains in the cell contaminating the phosphate concentrate. This poor performance of the cationic route was not a problem of the column itself but rather of the poor selectivity exhibited by the collector. Given its theoretical advantages it seems advisable to pursue this route with other cationic collectors.

THEORETICAL STUDIES.

Experiments carried out to characterize a negative bias column are discussed here. Additional tests in which a single size quartz fraction (14x20 mesh) was floated with Armac T at different feed rates showed that up to feed rates as high as 500 g/min the mineral is recovered as fast as it can be fed in the column. At higher feed rates (1000 g/min or more) it is observed that the rate of concentrate becomes smaller than the feed rate indicating loss of particles to the tailings or accumulation in the column. (See appendix)

Air Hold-up

Air hold-up is an important parameter for modelling and scale-up purposes. We determined the effect of variables such as air and water flowrates and frother concentration and the possible segregation of air hold-up in different zones of the column. The experimental method consisted in determining the difference of hydrostatic pressure between the column and a communicating vessel free of air bubbles. An alternative method based on the measurement of the void volume after suddenly stopping the flows of air and water was also used. Both methods gave similar results. Figures 14 summarize some results obtained in these determinations, additional data are given in the appendix. Main conclusions that can be drawn are as follows: There is an important relationship between air hold-up and air velocity. In the range of interest, the effect of frother concentration and water velocity is small. Adequate air hold-up is easily achieved even at the highest water velocity tested. There is also a small vertical segregation of air hold-up that could became a factor if a much taller column is used, however results presented above and also those in Chapter 1 indicate that a long column would not be necessary.

Residence Time Determinations

Residence time of corse phosphate particles in the 7 cm column were measured experimentally for various particle sizes. Typical results obtained for 14x20 mesh particles are given in Figure 15. A summary of the results for different sizes was presented in Chapter 1.

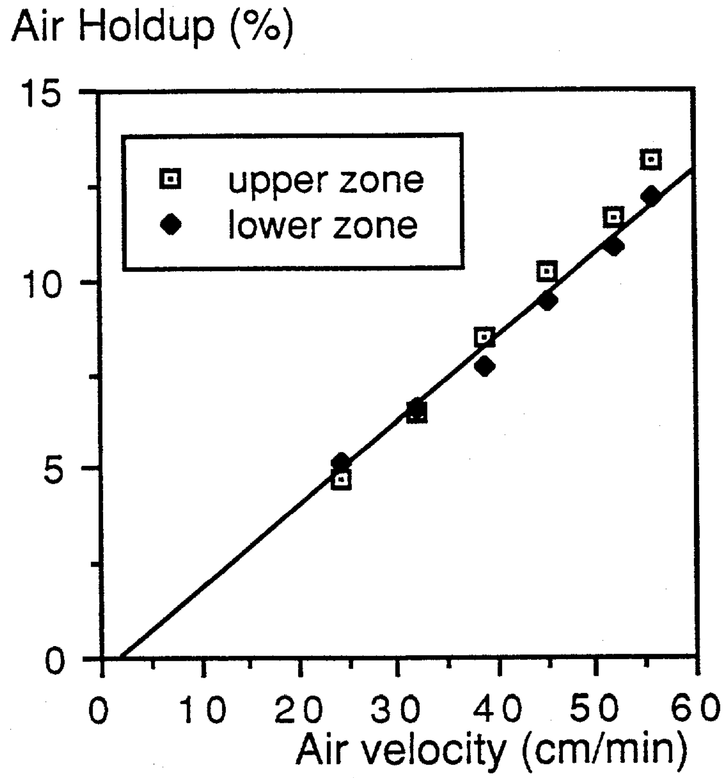

Figure 14. Air hold-up as a function of air velocity.

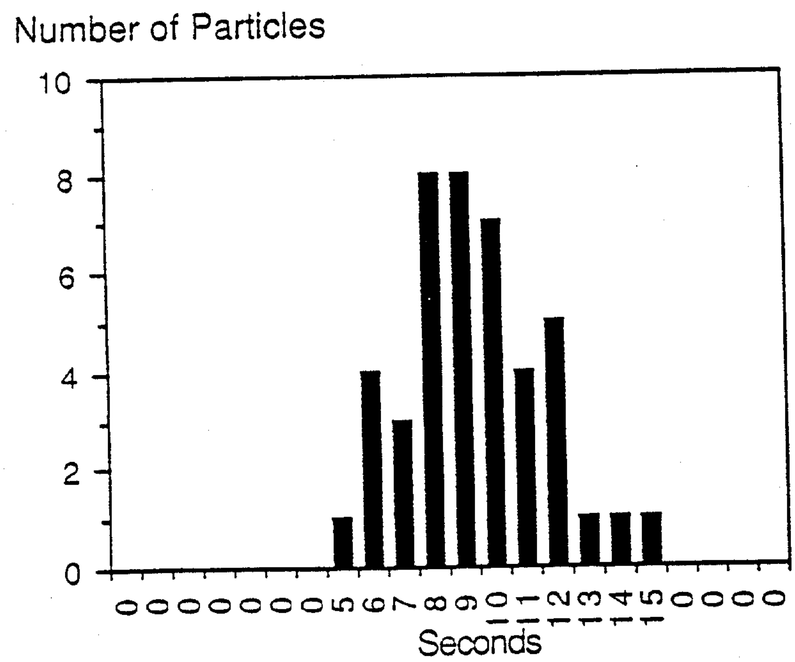

Figure 15. Settling time distribution for 14x20 mesh phosphate particles in 120 cm column.

FINAL REMARKS

Conventional flotation cells are required to perform two inherently contradictory tasks: first to provide enough agitation to create the turbulence level necessary to suspend the particles, disperse the air, promote the subsequent bubble-particle collisions and, secondly, to provide quiescent hydrodynamic conditions to avoid disruption of the particle-bubble aggregate and also avoid the transfer of gangue to the froth layer. With more or less ingenious designs of tanks, impellers and baffles these tasks can be accomplished fairly well by most mechanical cells when not too coarse particles (or not too fine) are treated.

The situation is different when flotation of coarse particles is intended, as shown in Chapter 3, a higher level of turbulence is required to keep the particles from settling, but at the same time, less turbulence is required to avoid disrupting particle-bubble aggregates, as a result performance of the cell is impaired. In the case of phosphate flotation, an additional disadvantage of mechanical cells is derived from the distinctive softness and tendency to disintegrate and form slimes exhibited by sedimentary phosphorites. The particle-particle interactions prevailing in mechanical cells are strong enough to produce abrasion of soft minerals, this attritioning should be cause of concern because may result in excessive reagent consumption and perhaps some loss of values. This effect is more noticeable in the case of softer ores such as dolomite contaminated phosphate rock that will be the typical feed in future mining in Florida.

Conventional flotation column have shown definitive industrial success in the flotation of molybdenite and zinc in Canada. Presently columns are being seriously considered for different tasks in countries worldwide. Indeed column flotation is one of the most active research and development areas in mineral processing. Although primarily used for flotation of fine particles due to effective control of fines entrainment by a wash water flow, columns have several advantages that can be considered of interest for any application including coarse particles no mechanical parts (therefore easy maintenance and low energy consumption), easy automatization and control, low turbulence, easy control of bubble size, simple flow pattern, well defined hydrodynamic conditions (therefore easier to model and scale up) and relatively high throughput. Since the flow pattern is plug-type there is no short circuiting of particles (or bubbles) and one column could replace an entire bank of cells.

Negative Bias Columns for Coarse Particles Flotation

Two column characteristics are extremely important for coarse particles flotation: a low turbulence regime and the possibility to control bubble size. The only factors that seem to keep conventional columns from being ideally suitable for coarse particles flotation are the wash water flow and the thick froth layer. However, as the experimental work discussed in Chapter 1 shows, the simple modification of using a net upwards flow of water (negative bias) countercurrent to the particles flow (instead of the co-current flow featured by conventional columns) could solve the problem. This type of column would maintain all the advantages of the flotation column mentioned earlier and would be more appropriate for the flotation of coarse feeds. A froth layer can be avoided and complete levitation of the particles is not be required, the drag force of the upwards water flow helps to levitate, or rather, elutriate the bubble-particle aggregates. This idea of using an upwards water flow has not been considered in the design of any flotation cell, but it has been applied implicitly when conventional flotation machines are operated (as is the usual practice in coarse phosphate and potash flotation) under “flooded condition” i.e. when the froth layer is practically inexistent in all but the first couple of cells of a flotation bank. Obviously, in highly turbulent mechanical cells this practice implies the transfer of gangue into the concentrated or froth product. In a column however, in a quasi-laminar flow, it was shown that the entrainment of fines during flotation of coarsely sized phosphate, could be kept at a minimum.

It was shown in Chapter 1 that flotation of coarse phosphate with anionic collectors in a negative bias column is extremely fast. A collection zone of less than one meter was enough to yield nearly 100 % BPL recovery with reasonable good selectivity. The process was no sensitive to particle size distribution when spiral feed, coarse flotation feed or unsized feed were used. A small upwards water flow (negative bias) was needed to transfer floating particles to the concentrate without a froth layer on top of the column. Bubble size is an important variable and consequently, those variables that directly or indirectly affect bubble size must be closely monitored and controlled. These variables include: frother type, frother concentration, aerator surface, pore size of the aerator, air flowrate and water flowrate around the aerator. However, a convenient size bubble was easily achieved with a simple and inexpensive aerator.

APPENDIX

Table 46. Anionic flotation of spiral feed in 7 cm column.

Conditioning time 3 minutes.

Test#	Tall oil kg/t	Fuel oil kg/t	Recovery %	Grade BPL %	Grade Insols %
1	1.0	2.0	3.4	59.8	20.6
2	1.6	2.0	30.2	61.6	19.8
3	2.5	1.25	42.1	60.5	19.9
4	2.5	3.7	93.8	58.9	20.5
5	2.5	5.5	99.8	58.6	21.8
6	2.0	1.0	28.6	60.5	20.1
7	2.0	3.0	92.1	61.0	18.1
8	2.0	2.0	48.1	58.9	21.2

Table 47. Anionic flotation of spiral feed in 7.0 cm column.

Conditioning time 8 minutes.

Test #	Tall oil Kg/t	Fuel oil kg/t	Recovery %	Grade BPL %	Insols %
30	2.0	6.0	99.6	56.0	24.0
31	2.5	5.5	99.8	55.0	26.0
32	2.0	4.0	99.5	57.4	23.1
33	1.5	3.5	99.4	61.9	17.5
34	1.2	3.0	99.3	61.8	17.5
35	1.0	3.0	99.2	61.7	17.7
36	0.9	2.7	92.1	67.3	10.2
37	1.0	2.0	62.0	67.0	11.0

Table 48. Anionic flotation of coarse flotation feed in 7.0 cm column.

Conditioning time 3 min.

Test #	Tall oil kg/t	Fuel oil kg/t	Recovery %	Grade BPL %	Insols %
9	1.0	2.0	10.1	56.1	26
10	1.5	2.0	21.2	58.5	22
11	2.0	2.0	45.2	58.2	22
12	2.0	3.0	87.6	59.4	20
13	2.5	2.5	91.0	59.6	20
14	2.0	2.0	80.5	60.8	19
15	2.5	2.5	95.5	58.7	22

Table 49. Anionic flotation of coarse flotation feed in 7.0 cm column.

Conditioning time 8 min.

Test#	Tall oil kg/t	Fuel oil kg/t	Recovery %	Grade BPL %	Grade Insols %
39	1.0	2.0	75.2	61.9	17.5
40	1.2	2.2	89.1	59.6	20.5
41	0.9	2.7	78.0	62.1	16.0
42	1.3	2.3	95.3	58.7	21.7
43	1.5	2.5	96.7	55.2	26.6
44	2.0	3.0	85.9	58.8	21.6
45	1.3	2.3	84.2	64.1	13.5

Table 50. Anionic flotation of mixture spiral and coarse flotation feeds in 7 cm column.

Test #	Tall oil kg/t	Fuel oil kg/t	Recovery %	Grade BPL %	Grade Insols %
47	1.3	2.3	98.7	60.0	20.0
48	1.3	2.3	87.0	64.6	13.9
49*	1.3	2.3	77.2	63.7	15.0
50	1.5	2.5	98.9	58.0	21.2
51	1.0	2.0	69.8	64.8	14.3
52	1.5	2.0	91.8	63.2	15.0

* Problems in pH control

Table 51 : Entrainment of fine quartz particles at different water flow rates.

MESH	FLOW RATE cm/s	O/F grams	U/F grams	ENTRAINMENT %
	22	0	250	0
	25	0	250	0
10/20	28	0	250	0
	22	0	250	0
20/28	25	0	250	0
	28	0.3	250	0.1
	22	0	250	0
28/35	25	0.5	249	0.2
	28	1.3	248	0.5
	22	3.0	247	1.2
35/65	25	6.3	244	2.5
	28	11.7	238	4.7
	22	4.3	246	1.7
65/100	25	7.8	242	3.1
	28	12.7	237	5.1
	22	6.2	244	2.5
-100	25	12.5	237	5.0
	28	18.5	231	7.4

Table 52: Quartz recovery as a function of rotor speed and particle size.

Mesh	rpm	grams floated	grams non floated	total	recovery %
	722	0.0	63.8	63.8	0.0
8 / 9	923	0.0	69.6	69.6	0.0
	1097	0.3	65.1	65.4	0.5
	722	0.8	102.2	103.0	0.8
9 /10	923	0.8	99.3	100.1	0.8
	1097	0.9	98.5	99.4	0.9
	722	3.6	94.9	98.5	3.7
10/12	923	2.9	97.2	100.1	2.9
	1097	3.2	99.4	102.6	3.1
	722	8.2	66.4	74.6	11.0
12/14	923	7.8	70.6	78.4	9.9
	1097	7.8	70.1	77.9	10.0
	722	31.6	91.8	123.4	25.6
14/20	923	34.2	94.7	128.9	26.5
	1097	31.6	94.9	126.5	25.0
	722	6.3	13.3	19.6	32.1
20/28	923	7.9	14.6	22.5	35.1
	1097	7.2	15.9	23.1	31.2
	722	86.1	67.9	154.0	55.9
28/35	923	98.6	55.7	154.3	63.9
	1097	93.7	63.8	157.5	59.5
	722	11.5	1.5	13.0	88.5
-35	923	22.3	6.0	28.3	78.8
	1097	24.5	7.7	32.2	76.1
	722	148.1	501.8	649.9	22.8
Overall	923	174.5	507.7	682.2	25.6
	1097	169.2	515.4	684.6	24.0