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EVALUATION OF A NOVEL CONTACT CELL FOR FINE PHOSPHATE RECOVERY

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Research Staff

Executive Director

Paul R. Clifford

Research Directors

G. Michael Lloyd, Jr.

J. Patrick Zhang

Steven G. Richardson

Gordon D. Nifong

-Chemical Processing

-Mining & Beneficiation

-Reclamation

-Environmental Services

Florida Institute of Phosphate Research

1855 West Main Street

Bartow, Florida 33830

(863) 534-7160

Fax: (863) 534-7165

<http://www.fipr.state.fl.us>

EVALUATION OF A NOVEL CONTACT CELL
FOR FINE PHOSPHATE RECOVERY

FINAL REPORT

Mku T. Ityokumbul
Principal Investigator

THE PENNSYLVANIA STATE UNIVERSITY
University Park, PA 16802

Prepared for

FLORIDA INSTITUTE OF PHOSPHATE RESEARCH
1855 West Main Street
Bartow, Florida 33830 USA

Contract Manager: Patrick Zhang
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PERSPECTIVE

Patrick Zhang, Research Director - Beneficiation & Mining

Phosphate mining in central Florida isolates approximately one ton of phosphatic waste clays for each ton of phosphate rock produced. Based on this ratio, approximately 1.5 billion tons of phosphatic clays have been generated by the Florida phosphate industry. In many cases, phosphate content in the phosphatic clays is higher than that in the matrix and therefore, about one-third of the phosphate mined has ended up in the clays. This enormous loss of phosphate has been repeatedly verified by many investigators.

The most dramatic development in the area of fine particle separation has been column flotation. A flotation column is a bubble column device that achieves separation between solid particles by using differences in their affinity for air bubbles. The concept of bubble column flotation was first developed and patented in the mid-1910s. However, industrial acceptance of flotation column cells was not realized until the early 1980s. At present, flotation columns are widely used in the mineral processing industry for such diverse separations as copper/molybdenum, copper/nickel, phosphate/gangue, and coal/refuse.

Column cells have been used primarily on coarse particles in Florida's phosphate beneficiation plants. The recovery of phosphate from the coarse flotation feed (typically 16 by 35 mesh) used to be a major efficiency problem. Separate flotation of this feed using mechanical cells could recover only about 60% of the phosphate; column flotation has brought that recovery up to 90%. Columns are also believed to be more suitable than mechanical cells for floating fine (below 150 mesh) feeds. This has been demonstrated in coal and metallic minerals processing. There are two potential applications for fine flotation technology in phosphate beneficiation. One is for the coarse fraction of the phosphatic clays, and another is for separating dolomite from the future dolomitic pebbles. Both applications are significant in terms of industry efficiency and resource conservation.

The issue of low throughput with flotation columns has been largely overcome with the development of the contact flotation cell. The heart of this new separation process is the turbulent contact chamber where particle-bubble contact is effected. As a result of the turbulent conditions in the contact chamber, particle collection is complete in about a second. Thus the flotation column is mainly used to effect the separation of the bubble-particle aggregates.

The contact column has two major advantages: fast collection that translates to high throughput; and quieter froth, which is beneficial to higher recovery from fine feed materials. Commercial application of the contact column has not been achieved yet. One of the main problems has been the lack of scale-up procedure. The principal investigator is one of the leading advocates of this novel column, and has been working on both scale-

up modeling of the column and testing on different minerals. This project demonstrated that selective flotation of phosphate from the bulk phosphatic clay is not feasible, suggesting that removal of the clay minerals is necessary for economic recovery of some of the phosphate values from phosphatic clays. What is left for study is how the contact column works on the coarser fraction, for example, the plus 400-mesh fraction.

ABSTRACT

Continuous experiments were carried out in a computer-controlled contact column flotation system to determine the feasibility of recovering the phosphate values in phosphatic clays. The contact column is an improvement over the conventional flotation column with significantly increased throughput. The heart of this new separation process is the turbulent contact chamber where particle-bubble contact is effected. As a result of the turbulent conditions in the contact chamber, particle collection is complete in about a second. Thus the flotation column is mainly used to effect the separation of the bubble-particle aggregates. The contact column has two major advantages: fast collection that translates to high throughput; and quieter froth, which is beneficial to higher recovery from fine feed materials. Flotation of a bulk phosphatic clay sample with a fatty acid collector showed no selection separation of phosphate from gangue materials (mainly clay minerals and sands).

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EXECUTIVE SUMMARY

Several processes have been developed and evaluated with FIPR funding for the recovery of the fine phosphate fraction that is currently lost. These losses, which may contain up to 30% of the phosphate value present in the plant feed, are the result of the present processing technology. In the current practice, the feed is deslimed at minus 150 mesh, and the phosphatic clay slimes impounded. Previous evaluations were carried out at the U.S. Bureau of Mines and the University of Florida. However, the recoveries and/or selectivity from these processes were not found to be economical. In view of the strategic importance attached to Florida Phosphate resource conservation and tailings, the beneficiation of these phosphatic clay slimes continues. Thus, the FIPR Board of Directors approved a one-year feasibility study (FIPR # 95-02-110) to evaluate the use of a novel contact column cell for this purpose.

Phosphatic clay slimes were collected from an operating IMC plant in Florida. The clay slimes were characterized and floated in the novel contact column cell. Size classification of the clay slimes was carried out in 25-mm hydrocyclones of varying underflow and overflow dimensions. Analyses of the underflow and overflow streams show that with the exception of the dolomite, the phosphate value and refuse (silica and clay minerals) were concentrated in the minus 5 μm size fraction. Thus, the dolomite content of the slimes can be reduced by classification. However, the by-pass fraction for these hydrocyclones was estimated from the size selectivity analysis to be in the range 20-40%. Under these conditions, fine phosphate losses to the underflow stream were in the range of 44-68%, classification of these slimes prior to flotation is not recommended.

Preliminary batch flotation experiments were carried out to determine the factors that affect the separation. Analysis of the results shows that the phosphate and mineral recoveries were approximately identical. However, there was a slight increase in the dolomite content of the froth product. This finding suggests that the primary mechanism for solids recovery was entrainment. Owing to the fine nature of the feed material ($d_{80} < 10 \mu\text{m}$), this finding is reasonable. Thus, the remaining flotation tests, were carried out in our computer-controlled 0.076 m x 3.55 m flotation column.

Continuous experiments carried out in a computer-controlled pilot plant flotation column confirmed that batch flotation results. In these tests, wash water was added at the rate of 1.2 liters/min to suppress gangue recovery. While wash water addition helped to reduce the gangue entrainment, it also resulted in a lowering of the phosphate recovery. By contrast, the M_6O content of the froth product was increased. This finding suggests that without size enlargement (e.g. selective flocculation), fine phosphate recovery from these clay slimes cannot be accomplished with the proposed method. These results suggest that entrainment was the predominant mechanism for solids recovery during flotation. Thus, it appears that these slimes are below the limit of economic recovery for the phosphate value by traditional surface-based separation techniques. It is recommended that other separation techniques (e.g. aqueous bi-phase separation, flocculation, etc.) be exploited in conjunction with the novel contact column cell.

INTRODUCTION

In the Florida phosphate industry, the feed to the froth flotation circuit is deslimed at 150-mesh (105 μm) using hydrocyclones. The hydrocyclone overflow, which contains the phosphatic clay slimes is sent to tailings ponds as a dilute slurry (3-5% solids) (US Bureau of Mines 1975; National Research Council 1979; Zhang 1993). While the actual phosphate content of the tailing slimes varies, it typically contains about 30% of the total phosphate present in the plant feed (Zhang 1993). At the current production levels, it is estimated that over 3 million tons of phosphate (expressed as P_2O_5) are discarded with the phosphatic clays in Florida each year (Zhang 1993).

The phosphate production operation generates phosphatic clay slimes that are approximately 25% larger than the volume of mined phosphate matrix (Boyle 1969). This volume increase is due to the poor settling and compaction characteristics of the clay slimes. Thus, the safe disposal of the waste phosphate slimes presents an important problem in the Florida phosphate rock production. The current disposal practice involves storage of the phosphatic clay slimes in dams specially constructed around mined out areas. However, the direct impoundment of the phosphatic clay slimes is environmentally inadequate due to the possibility of dike failure during times of heavy precipitation (e.g., the Tesoro dam failure in Italy on July 19, 1985). For this reason, the continued exploitation of the Florida phosphate deposits may depend on the development of processes that will provide for the environmentally acceptable:

- reclamation of the dikes;
- reclamation of the phosphatic clay slimes (including recovery of fine phosphate therefrom), and
- minimization of fresh water requirements with the adoption of process water recovery and recycling practices.

It is noted that by-product recovery from the slimes may have a significant impact on the economics of tailings treatment as has been demonstrated for synthetic oil industry in Northern Alberta (Ityokumbul et al. 1987; 1994).

The nature and characteristics of the phosphatic clay slimes present unique processing challenges. For example, the slimes are extremely fine (30-35% less than 1 μm), with the clay minerals comprising nearly 50% of the total weight. Furthermore, the slimes are contaminated with dolomite and hematite. Similarly, over 70% of the phosphate value is contained in the minus 20 μm fraction (Jordan et al. 1982; Davis et al 1987). Therefore, new processing techniques must be developed to recover the fine phosphate value from these slimes.

LITERATURE REVIEW

CONVENTIONAL FLOTATION

The recovery of phosphate from phosphatic clay slimes by froth flotation has been studied by a number of researchers (Jordan et al. 1982; Davis et al. 1987; Singh et al. 1992). Jordan et al. (1982) and Davis et al. (1987) deslimed tailings material from Florida phosphate operations using hydrocyclones and hydroseparators to remove the very fine fractions (minus 37 μm or minus 20 μm) prior to flotation. By using two stages of cleaning in a conventional flotation cell, concentrates of 21 to 33.4% P_2O_5 were obtained at recoveries of 68 to 88%. As indicated earlier, over 70% of the phosphate value in these slimes is present in the minus 20 μm fraction. This means that the overall phosphate recovery from the clay slimes was less than 30%.

Singh et al. (1992) reported that a Maton (India) phosphate slimes containing 17% P_2O_5 could be upgraded by conventional flotation to approximately 30% P_2O_5 at about 90% recovery. The silica content of the phosphate concentrate was high (15%) even though the sodium silicate dosage was much higher than that used in the plant flotation. Surprisingly, the collector dosage required for this separation was lower than that used in the plant operations. Similarly, Prasad et al. (1995) have used flotation to successfully recover fine phosphate (82% less than 75 μm) from cherty-calcareous rock phosphate ore. However, owing to the high silica content of the concentrate, reverse flotation of the silica was necessary. The high silica content of the concentrate was attributed to entrainment of the fine silica particles.

COLUMN FLOTATION

The most dramatic development in the area of fine particle flotation has been column flotation. A flotation column is a bubble column device that achieves separation between solid particles by using differences in their affinity for air bubbles. Unlike conventional flotation cells, mixing in a flotation column cell is induced by the passage of air bubbles. This produces quiescent mixing conditions that are required for fine particle recovery. The concept of bubble column flotation was first developed and patented in the mid-1910s by Flinn and Towne (Gahl 1916). However, industrial acceptance of flotation column cells was not realized until the early 1980s. At present, flotation columns are widely used in the mineral processing industry for such diverse separations as copper/molybdenum, copper/nickel, phosphate/gangue, and coal/refuse.

The use of column cells in the phosphate industry has focused primarily on the recovery of coarse (+48 mesh) phosphate particles (Hollingsworth 1967; Soto and Barbery 1991, etc.). Quite recently, Hutwelker et al. (1993) have reported the flotation of 20x200 mesh North Carolina phosphate in a short column. Indeed, the use of short columns is supported by the author's work (e.g., see Ityokumbul 1986; 1992; 1993; etc.) and is consistent with the first column design (Gahl 1916). However, the particle sizes

tested by Hutwelker et al. (1993) were considerably larger than those found in a typical Florida phosphatic clay slime. Thus, the applicability of their findings to the recovery of phosphate from the fine phosphatic clay slimes cannot be ascertained at the present time.

Harris et al. (1994) compared the performance of different flotation devices (conventional, column, air-sparged hydrocyclone [ASH], and Jameson cells) for fine coal cleaning. Their results indicated that column flotation gave the highest yields for fine (minus 25 μm) and intermediate (150x25 μm) size fractions. However, a major limitation of column cells has been the low throughputs, which was reported to be between one and two orders of magnitude lower than the ASH cell.

The issue of low flotation column throughputs has been largely overcome with the development of the contact column flotation cell in Canada (Amelunxen 1994). The heart of this new separation process is the turbulent contact chamber where particle-bubble contact is effected. This is accomplished by contacting the slurry with air bubbles from a USBM-type bubble generator in a turbulent chamber. As a result of the turbulent conditions existing in the contact chamber, particle collection is complete in about 1 second. Thus, the flotation column is mainly used to effect the separation of the bubble-particle aggregates from the slurry. It is noted that high recoveries were observed for both coarse and fine size fractions in Cu/Mo separations.

In order to recover the fine phosphate mineral particles present in these slimes, it is essential to generate fine bubbles. Ongoing studies at Penn State University suggest that small bubbles may be generated with a new turbulent contact bubble generator. Unlike the original contact cell, our device does not require a separate unit for bubble generation. This work, supported by an Engineering Foundation Initiation Grant (RI-A-94-03), has allowed us to determine the optimum operating conditions for generating fine bubbles. With frother addition, we are presently capable of generating 0.5 – 1 mm bubbles. Since maintaining quiescent conditions is essential for recovering fine particles in the contact column cell, a characterization of the turbulence levels within the column cell was also carried out (Ityokumbul 1995).

FINE SIZE CLASSIFICATION

One of the keys to recovering the fine phosphate minerals by flotation is desliming. Since the separation efficiency of flotation will invariably decrease at very fine sizes (e.g. see Jordan et al. 1982), it will be necessary to remove the ultrafine particles to improve the selectivity of the overall separation process.

The most commonly used device for classifying particles at fine sizes is the hydrocyclone. Separations down to about 10 μm are possible with this device. However, since the cut size is related to the diameter of the hydrocyclone, classification at fine sizes requires a small diameter hydrocyclone. For example, classification at 10 μm would likely require the use of a 25-mm diameter unit. Moreover, since hydrocyclone capacity

is proportional to (cyclone diameter)², the finer cut sizes are achieved at the expense of volumetric throughput.

Alternatively, it should be possible to separate at sizes of 10 μm and finer using a solid-bowl (decanter) centrifuge. This device offers at least one potential advantage over the hydrocyclone; the ability to control independently the separation times (by changing the feed rate) and the number of g's (by changing the bowl speed). This approach should allow finer particles to be separated, while still operating at reasonable feed rates. These devices have been used to classify particles finer than 10 μm in various mineral applications (Williamson and Bacon 1977; Scheffler and Zahr 1980).

EXPERIMENTAL

EQUIPMENT

Preliminary flotation experiments were carried out using unclassified feed material to establish baseline-testing conditions. The semi-batch and continuous flotation experiments were carried out in our 0.064 m x 2.1 m and computer controlled 0.076 m x 3.55m flotation columns, respectively. The 0.076 m flotation column was equipped with the novel (turbulent) bubble generator. The operation of this new bubble generator entails contacting the air with the slurry in a turbulent contact chamber where the particle-bubble collision and contact is effected. The flotation column is used mainly to separate the bubble-particle agglomerates from the slurry. A schematic of the novel turbulent bubble generator is shown in Figure 1. In all of these continuous tests, the chamber pressure was fixed at 140 kPa (20 psi) which was previously determined to produce fine bubbles (Ityokumbul 1995).

MATERIALS

Four samples of the phosphatic clay slimes were collected from an IMC phosphate plant in Florida. Three of the samples were collected directly from the primary hydrocyclone overflow into plastic-lined 55-gallon containers. The solid content of these samples varied from 1.6-4.6 wt. %. The last sample was collected over a period of time by a process of repeated decantation. This sample contained about 28% solids by weight and may be considered to represent consolidated tailings material. The mineralogical composition of the solids were determined using a Rigaku x-ray diffractometer. The scan was from 5° to 75° and phase identification was done using a computer routine. In order to characterize feed, the particle size distribution of the samples was determined using a SPA Microtrac (Model X-100). A typical x-ray diffraction pattern and particle size distribution of the feed material is shown in Appendix A.

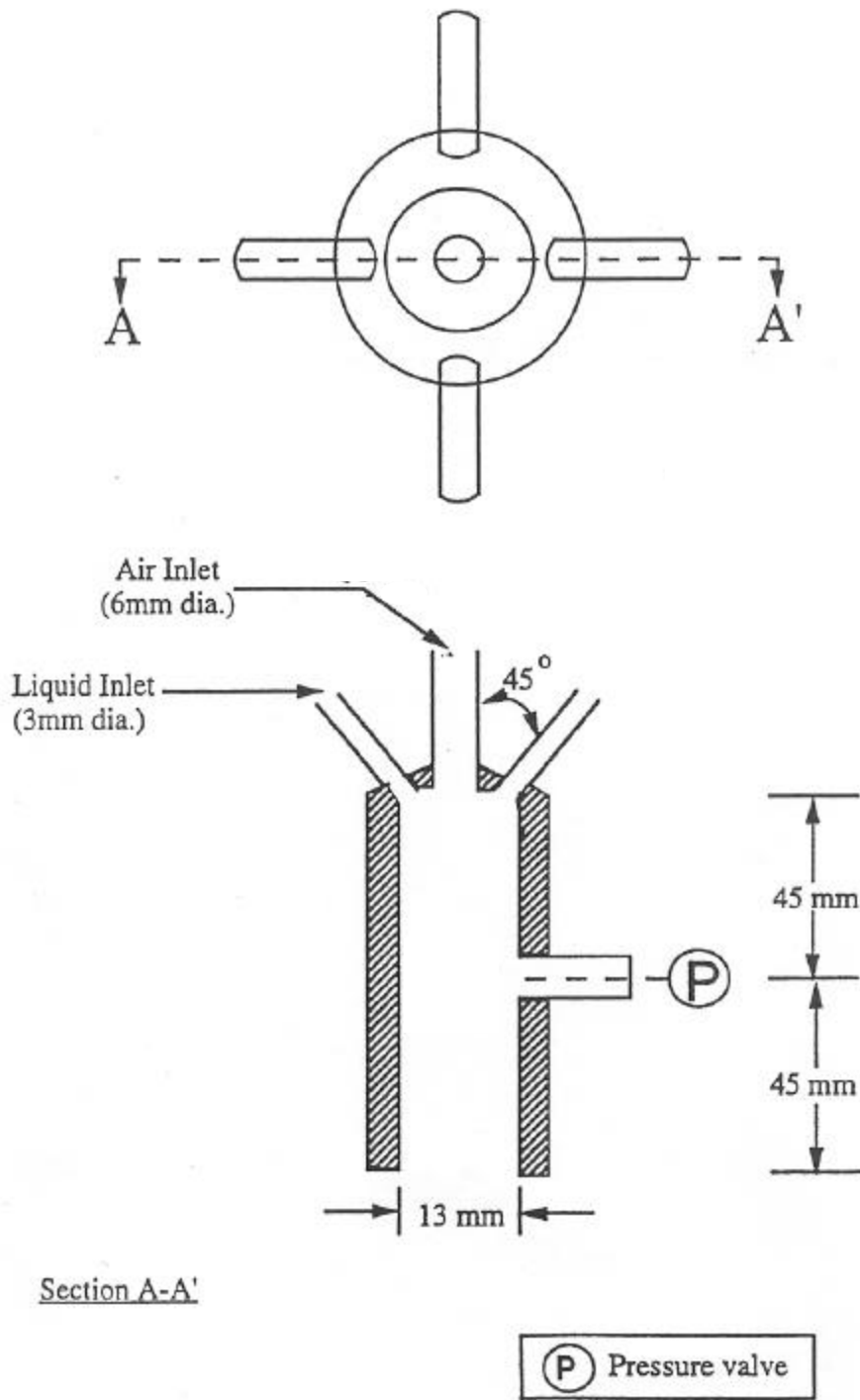


Figure 1. Detailed Drawing of the Vortactor Turbulent Contact Bubble Generator.

CLASSIFICATION

The partially consolidated phosphatic clay sample (S-525) was diluted to a solid concentration of 7-10% solids and classified in a 25-mm hydrocyclone. Preliminary experiments were carried out to determine the best geometry for the classification studies. In these tests, the underflow and overflow openings were varied. From the metallurgical balance, the promising ones were selected for more detailed study. Two hydrocyclone openings (3 mm and 5.5 mm) and two underflow openings (2.2 and 3.2mm) were used in the subsequent studies. Table 1 shows the settings used in the hydrocyclone tests. In all of these tests, the pressure drop was fixed at 349 kPa (50 psi). The quantities of solids and water reporting to the underflow and overflow streams were determined. From the composition of the solids reporting to the overflow and underflow streams, the relative distributions of the different components were determined.

Table 1. Hydrocyclone Settings and Feed Composition Used in the Classification Studies.

Parameter	Run 1	Run 2	Run 3
Overflow opening, mm	3.0	3.0	5.5
Underflow opening, mm	2.2	3.2	3.2
Feed rate, l/min	12.0	12.2	22.0
Feed concentration, wt. %	7.21	6.96	10.1
P ₂ O ₅ (wt. %)	7.69	8.04	7.88
MgO (wt. %)	4.85	5.08	5.74
CaO (wt. %)	15.7	16.4	17.3
SiO ₂ (wt. %)	33.1	32.0	30.9
Al ₂ O ₃ (wt. %)	13.7	14.0	12.7
Fe ₂ O ₃ (wt. %)	2.94	2.92	2.86

FLOTATION EXPERIMENTS

For the preliminary studies, batch flotation experiments were carried out. In these tests, the required reagents were added to approximately 4 liters of the unclassified feed material in a plastic bucket to which a laboratory mixer was attached. The required amount of sodium silicate was added to the conditioning vessel and mixing continued for 10 minutes. Thereafter, the collector was added and agitation continued for another 10 minutes. The conditioned feed was next transferred to the batch column where 20 PPM of Dowfroth 250C frother was added. However, when Custofloat AR-27 (a proprietary collector from Westvaco) was used, no additional frother was added. After circulation of the feed for 5 minutes, aeration was turned on and the material overflowing the cell lip was collected.

For the continuous flotation experiments, approximately 100 liters of the unclassified feed was required. This was prepared by diluting the partially consolidated sample (S-525) with water or mixing it with the dilute samples of the primary hydrocyclone overflow collected from the IMC plant. Owing to the high specific area of the feed solids, the solid concentration in the flotation feed was limited to approximately 3 to 5 wt. %. The Procedure for reagent addition described above for the batch flotation tests was followed with the exception that the frother was added directly to the slurry reservoir.

As indicated above, the continuous flotation experiments were carried out in our computer-controlled 0.076 m x 3.55 m pilot flotation column. Figure 2 shows a schematic of the experimental set-up for the continuous flotation testwork. The flotation column was fitted with six high-accuracy pressure transmitters (Cole-Palmer H-68971 series) at varying axial locations. The outputs from the sensors were fed to a 386 SX Everex computer at a frequency of 0.2 Hz. The desired hydrostatic head in the column (measure of the liquid level) was set using the pressure sensor located at the bottom of the column. The column was filled with slurry until the set hydrostatic head was reached. Thereafter, the air was turned on and carefully adjusted to the desired value. By regulating the tailings pump using an icon-based control program (COLUMNCT.VI), the hydrostatic head was maintained at the set value. The flotation column control program was developed using Labview Student Edition, provided by National Instruments.

For all the continuous flotation experiments, the novel turbulent bubble generator under development at Pennsylvania State University was used. As indicated above, the slurry feed and air were brought into intimate contact inside the Vortactor chamber where the pressure was set at approximately 140 kPa. A detailed description of the Vortactor turbulent contactor and its operation can be found elsewhere (Ityokumbul et al. 1996). The slurry-air mixture was introduced into the column at the desired axial location. Collection of timed froth and tailing samples were initiated the moment the froth overflowed the cell lip. For the froth samples, collection was over 30-second intervals for the first minute and 1 minute intervals for the remainder of the experimental run.

Previous studies in the unit (Ityokumbul and Trubelja 1998) had shown that froth removal would present a significant bottleneck in the accurate determination of the concentrate flow rate. This problem was overcome with the installation of a water ring spray on the outer periphery of the cell lip. For all the experiments reported here, the wash water addition rate when needed was added at the rate of 1.2 liters per minute (LPM). Figure 3 shows the variation of concentrate flow rate, weight percent solid recovery and percent solids in the tailings with flotation time. The results clearly show that steady state conditions are achieved in about 2-4 min. Subsequently, this mode of operation was adopted for all the flotation runs reported here.

The froth and tailings samples were filtered, dried and weighted. For the tailings samples, the amount of water was also measured in order to determine the tailings flow rate at any given time interval, and the per cent solids in the tailings stream. All of these

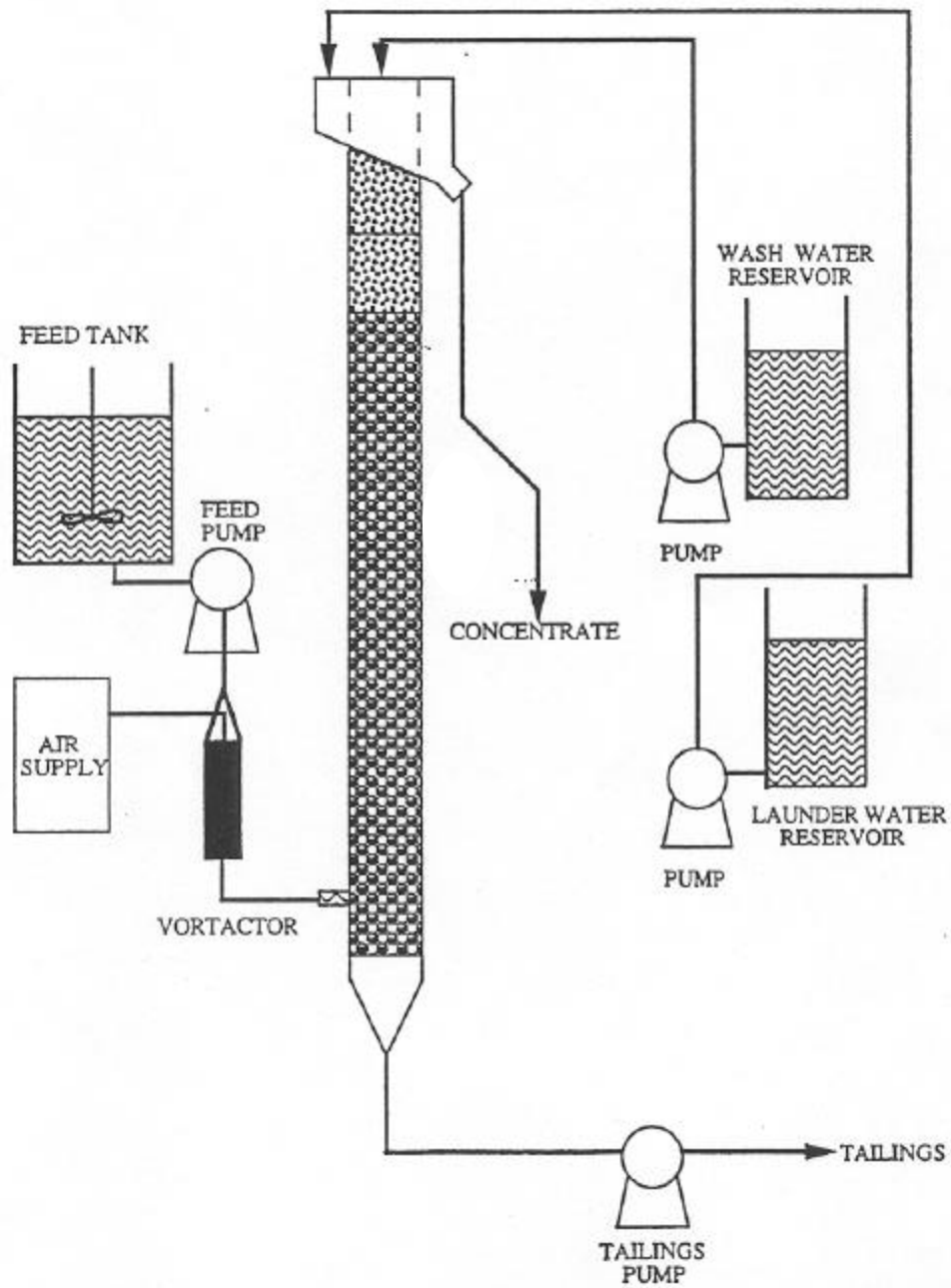


Figure 2. A Simple Schematic of the Column Flotation Pilot Plant Fitted with the Vortactor Sparger.

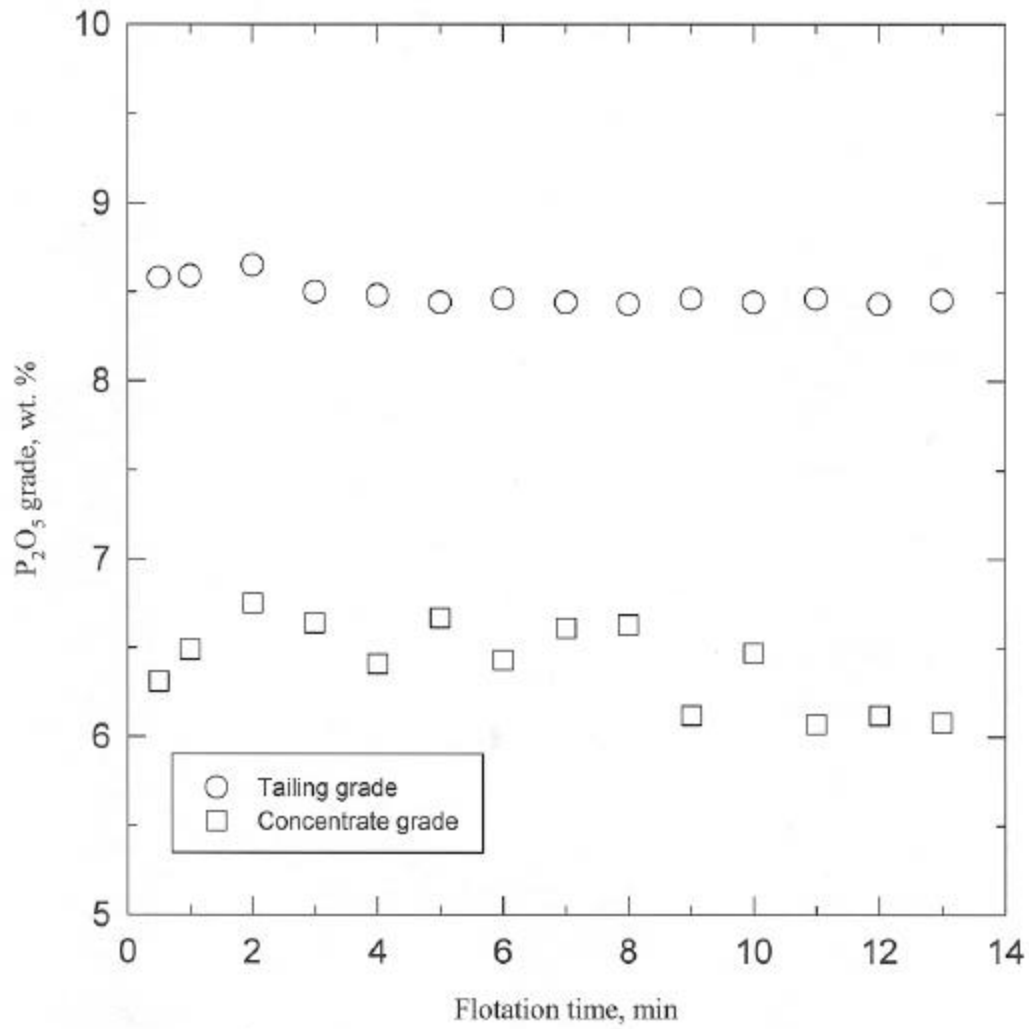


Figure 3. Variation of Concentrate and Tailings Grade with Continuous Flotation Time.

values were used to determine the attainment of steady state in our experiments (see Figure 3).

For the analysis of the feed, froth and tailing samples, a lithium metaborate fusion technique was used to dissolve the inorganic solids (see Appendix B for details). The concentration of cations in the solution was determined by inductive coupled plasma (ICP) spectrophotometer (Leeman Labs Model PS 3000UV).

As indicated earlier, static pressure measurements along the column height were recorded over the entire duration of the experimental run. These values were used to compute the average gas hold-up in the recovery zone. Using the drift flux approach, the average bubble size in the recovery zone was estimated (see Ityokumbul et al. 1995). With this information, the ratio of projected particle to bubble surface area was calculated for the different experimental runs.

GRANULOMETRIC SEPARATION

In addition to the flotation tests described above, the efficacy of granulometric separation was also evaluated. For this evaluation, Dowfroth 250C was added to the slurry and the mixture transferred to the batch column. Aeration was turned on and the material overflowing the cell lip was collected. The particle size distribution and composition of the float product and sink were determined.

RESULTS AND DISCUSSION

SAMPLE CHARACTERIZATION

The particle size distribution and composition of the phosphatic clay samples are shown in Table 2. The results confirm that the phosphatic clay samples are extremely fine in nature and are consistent with previous analysis (e.g. see Jordan et al. 1982; Davis et al. 1987; Zhang 1993). Since the first three samples represent the instantaneous samples from the overflow from the primary hydrocyclones, they suggest variation in the feed and/or efficiencies of the different hydrocyclones. Since 90% of the sample is below 400 mesh, the recovery of phosphate values from these slimes does present unique processing challenges.

GRANULOMETRIC SEPARATION

Table 3 shows the particle size distribution of the feed, float and sink material, and the relative distribution of the minerals from granulometric separation. For this test, the phosphatic clay sample used was S-390 and the air flow rate was fixed at a superficial velocity of 1.45 cm/s. As expected, the float product from the granulometric separation was finer than the sink material. However, the relative change, composition and composition of the products (see Table 3) did not justify the use of the technique. This finding was expected in view of the fine nature of the feed material and the distribution of the phosphate value in the sample. In the light of this finding, no further evaluation of the granulometric separation is suggested.

Table 2. Characteristics of the Phosphatic Clay Samples.

Parameter	Sample ID			
	S-360	S-390	S-455	S-525*
d_{10} , μm	1.4	2.1	1.9	1.7
d_{50} , μm	6.7	11.3	6.0	6.1
d_{90} , μm	28.5	37.2	19.6	23.3
Solids (wt. %)	1.63	4.64	1.61	28.5
P_2O_5 (wt. %)	8.35	7.43	5.47	6.89
MgO (wt. %)	3.73	5.12	5.79	5.42
CaO (wt. %)	14.4	14.2	13.0	16.5
SiO_2 (wt. %)	29.5	31.5	32.6	31.8
Al_2O_3 (wt. %)	18.1	15.6	14.3	14.4
Fe_2O_3 (wt. %)	2.44	2.88	3.24	2.86

* Partially consolidated samples produced from repeated decantation of clay slimes.

Table 3a. Characteristics of Granulometric Separation Products.

Parameter	Sample ID		
	Feed*	Float	Sink
d ₁₀ , μm	1.1	0.9	1.0
d ₅₀ , μm	5.1	4.0	4.9
d ₉₀ , μm	18.0	14.3	17.7

Table 3b. Mineral Distribution from Granulometric Separation of the Phosphatic Clay Slimes.

Parameter	Grade			Distribution, wt. %	
	Feed*	Float	Sink	Float	Sink
Solid distribution (wt. %)	100	41.3	58.7	41.3	58.7
P ₂ O ₅ (wt. %)	7.57	7.78	7.43	42.4	57.6
MgO (wt. %)	5.24	4.89	5.49	38.5	61.5
CaO (wt. %)	14.6	14.3	14.38	40.5	59.5
SiO ₂ (wt. %)	31.6	32.1	31.3	42.0	58.0
Al ₂ O ₃ (wt. %)	15.9	16.3	15.6	42.3	57.7
Fe ₂ O ₃ (wt. %)	2.94	2.98	2.91	41.9	58.1

* Computed composition of the feed material

CLASSIFICATION

Figures 4 through 6 show the partition curve for the hydrocyclones used in this study. The relative amounts of solids and water reporting to the underflow (UF) and overflow (OF) streams were determined. The results show that the amount of bypass varied from about 20% to a maximum of 40%. In general, the amount of bypass increased with underflow size. Table 4 shows the relative distribution of the different components in the underflow and overflow streams.

The results show that the behavior of SiO_2 , P_2O_5 and Fe_2O_3 were approximately the same and approximately equal to the relative distribution of the solids in the two streams (see Figure 7). In general, the concentration of these components (i.e. SiO_2 , P_2O_5 and Fe_2O_3) were slightly higher in the overflow stream thus suggesting that there were present in the finer fraction. Similarly, the distribution of the Al_2O_3 was considerably higher than the relative weight distribution of the streams. Since the concentration of Al_2O_3 in the overflow was consistently much higher than that in the underflow, it appears to suggest that it was predominantly present in the very fine fraction. The high values of SiO_2 and Al_2O_3 in the overflow are also suggestive of the presence of clays.

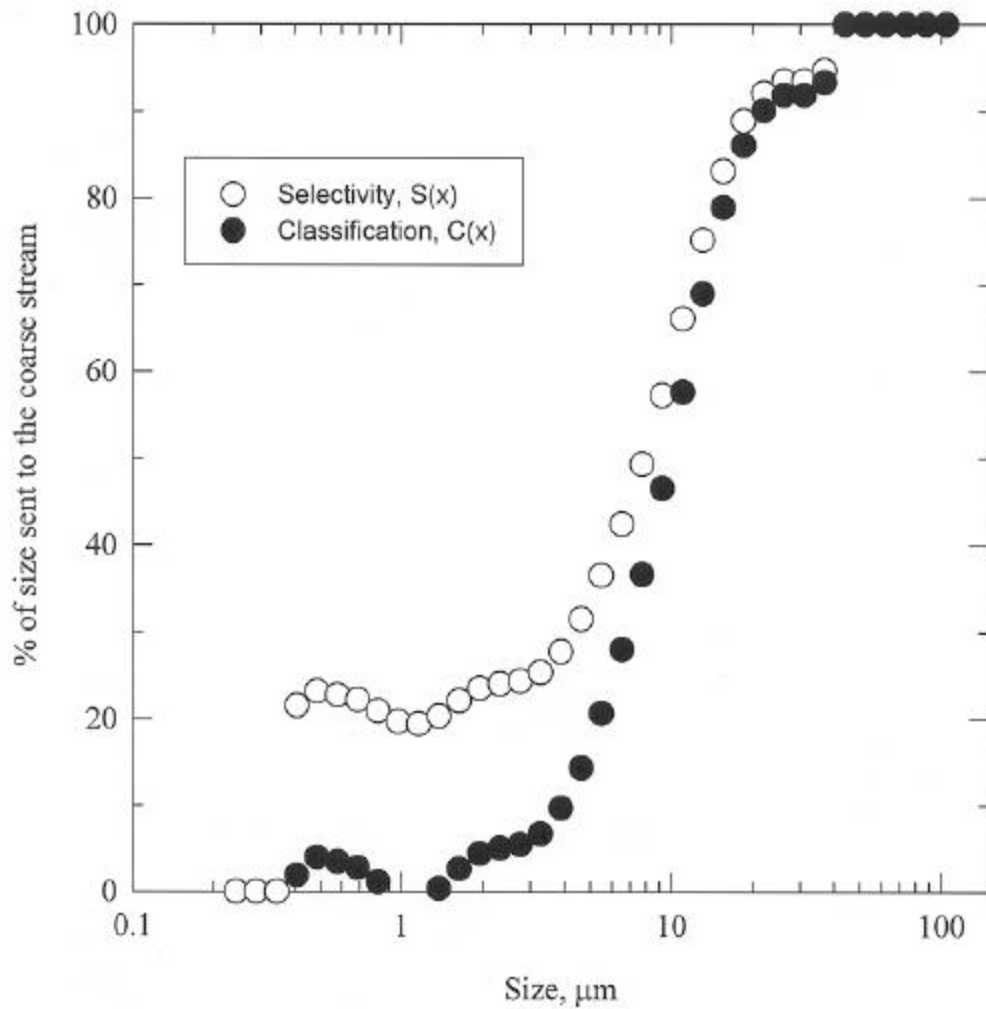


Figure 4. Classifier Selectivity Partition Curves for Run 1.

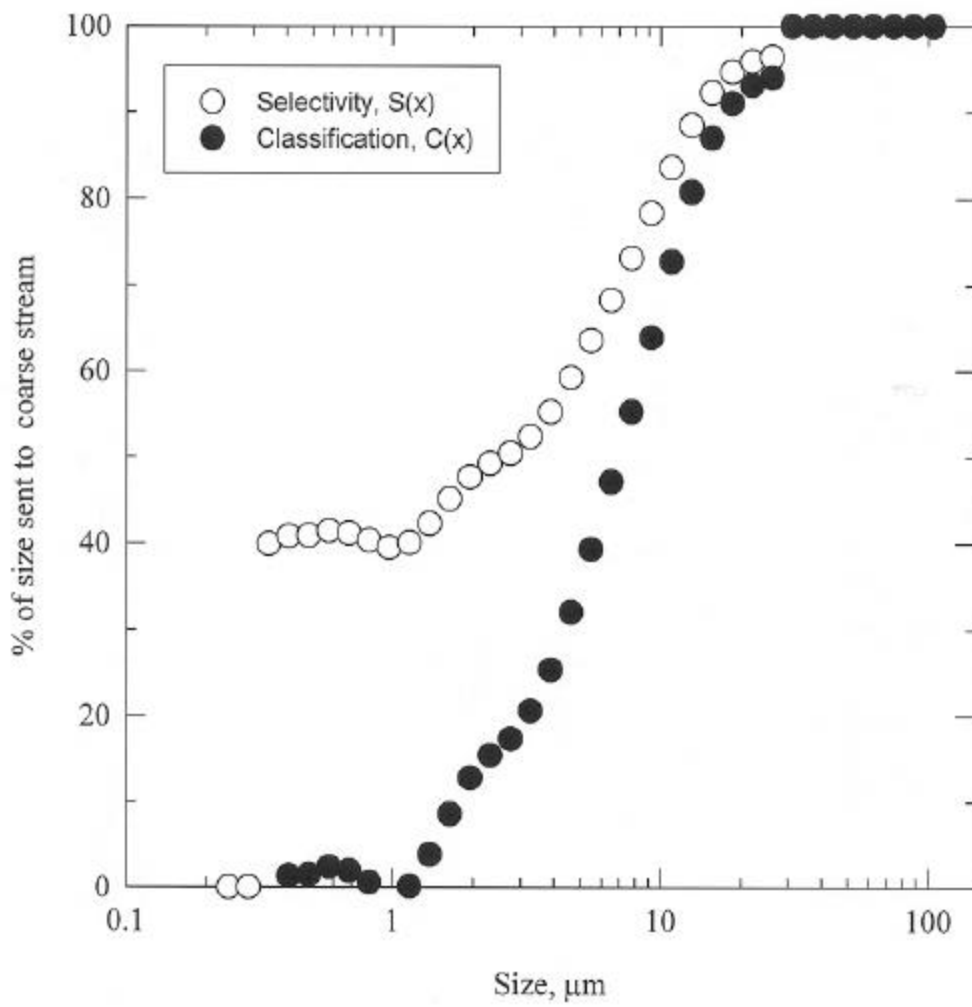


Figure 5. Classifier Selectivity Partition Curves for Run 2.

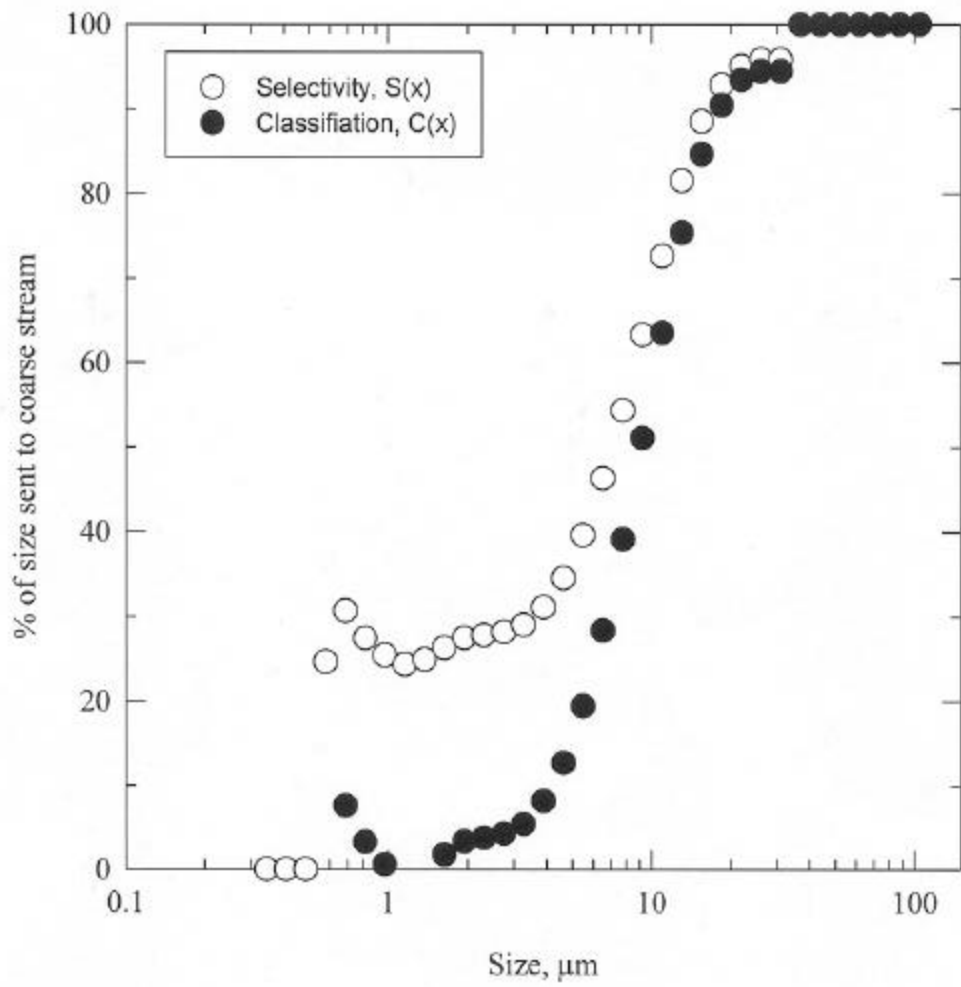


Figure 6. Classifier Selectivity Partition Curves for Run 3.

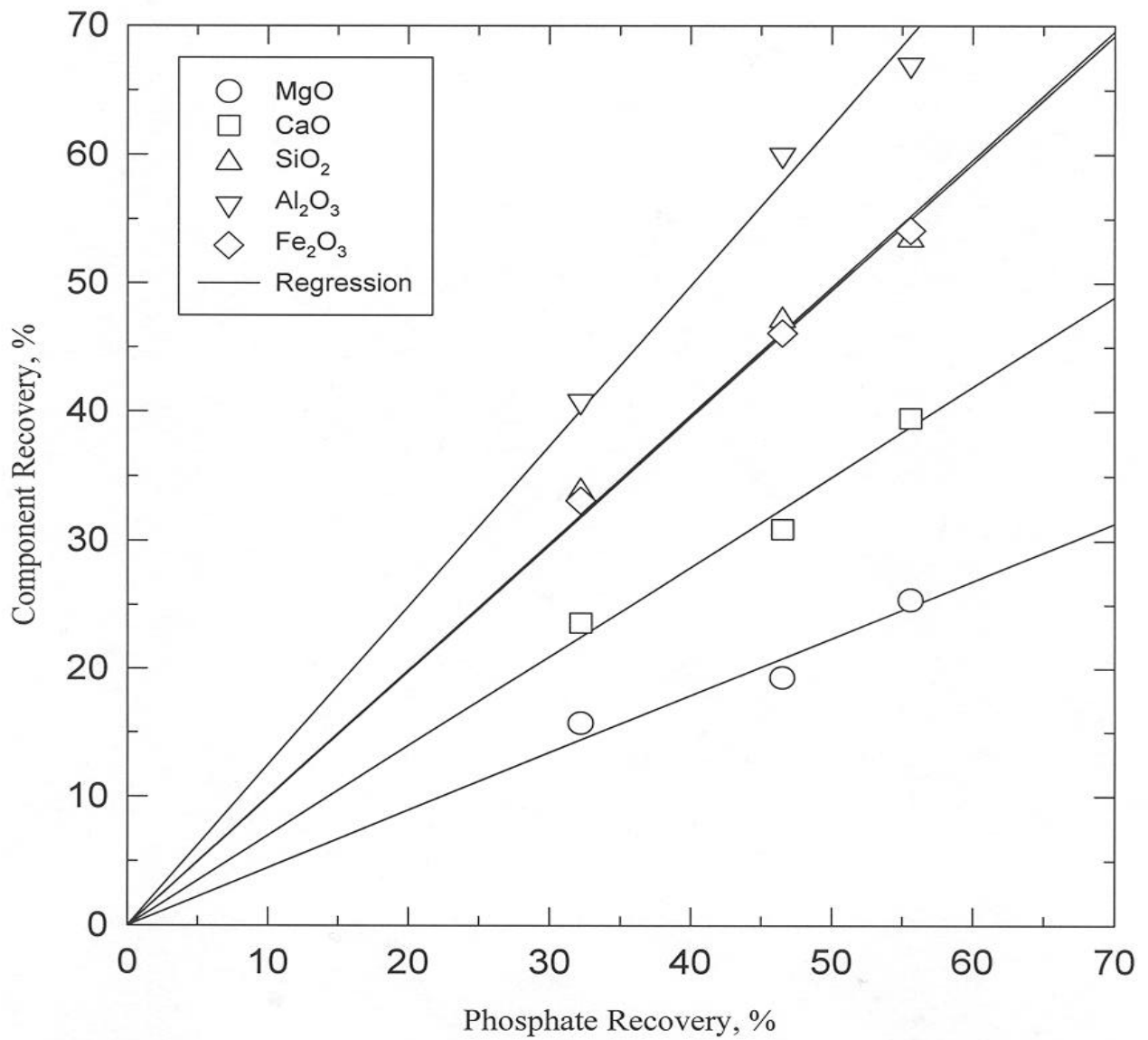


Figure 7. Selectivity of Phosphate Recovery from Phosphatic Clay Slimes in 25 mm Hydrocyclones.

Table 4. Performance Results from Classification of the Phosphatic Clay Slimes.

Parameter	Run 1		Run 2		Run 3	
	OF	UF	OF	UF	OF	UF
Volume distribution, %	71.4	28.6	44.8	55.2	79.5	20.5
Solid distribution, %	49.3	50.7	31.0	69.0	41.4	58.9
Solid Concentration, wt. %	4.97	12.8	4.82	8.69	5.20	29.0
d_{10} , μm	0.75	2.54	0.72	1.82	0.98	3.10
d_{50} , μm	3.35	11.5	2.96	8.24	3.70	12.4
d_{90} , μm	9.37	33.7	8.10	30.0	9.31	33.1
<u>Analysis</u>						
P ₂ O ₅ (wt. %)	8.67	6.74	8.62	7.78	8.90	7.16
MgO (wt. %)	2.50	7.14	2.58	6.21	2.69	7.86
CaO (wt. %)	12.6	18.8	12.5	18.2	13.0	20.3
SiO ₂ (wt. %)	35.8	30.4	34.9	30.7	35.4	27.8
Al ₂ O ₃ (wt. %)	18.6	8.90	18.4	12.0	18.5	8.61
Fe ₂ O ₃ (wt. %)	3.22	8.66	3.11	2.83	3.21	2.62
<u>Distribution</u>						
P ₂ O ₅ (wt. %)	55.6	44.4	32.2	66.8	46.5	53.5
MgO (wt. %)	25.4	74.6	15.7	84.3	19.3	80.7
CaO (wt. %)	39.5	60.5	23.6	76.4	30.9	69.1
SiO ₂ (wt. %)	53.4	46.6	33.8	66.2	47.1	52.9
Al ₂ O ₃ (wt. %)	67.0	33.0	40.8	59.2	60.0	40.0
Fe ₂ O ₃ (wt. %)	54.1	45.9	33.1	66.9	46.1	53.9

By contrast, the concentration of MgO and CaO were considerably higher in the underflow stream. This would suggest a preferential concentration of the dolomite in the coarse fraction of these samples.

The results from these classification studies show that to recover a reasonable portion of the phosphate present in the clay slimes the overflow and underflow streams would have to be processed separately. As an example, the overflow material, which contains a lower concentration of dolomite, might be flocculated prior to flotation. While underflow material has a size distribution that is ideal for column flotation, the separation of dolomite from the phosphate value does present a serious challenge. Since discarding of any of the fractions would result in high phosphate losses, the slimes from this particular plant were processed without classification.

BATCH FLOTATION EXPERIMENTS

In order to explain the changes taking place during conditioning, samples of the slurry were taken for particle size analysis. Table 5 shows the feed, conditioned and sink material particle size distribution.

Table 5. Variation of Particle Size Distribution with Dispersant and Collector Addition.

Parameter	Sample ID		
	Feed	Conditioned*	Sink
$d_{10}, \mu\text{m}$	1.3	1.0	1.0
$d_{50}, \mu\text{m}$	5.2	5.1	4.3
$d_{90}, \mu\text{m}$	17.6	24.9	13.7

* 4 liters of S-390 conditioned with 1 g sodium silicate followed by 2 g of sodium oleate at pH of 9.2

The results show that addition of sodium silicate promotes dispersion of the feed material. However, addition of the collector does appear to promote flocculation of the feed. For example, the d_{90} of the conditioned feed increased from about 18 to 25 μm . The results also show that flotation resulted in a preferential recovery of the coarse fraction as the sink had a finer particle size distribution relative to the conditioned material. From our classification studies, this would suggest enrichment in the MgO and CaO content of the froth product.

The conditioned feed was floated in the batch column. The weight recovery and composition of the float and sink streams are given in Table 6. The beneficial effect of sodium silicate is reflected in the lower SiO_2 and Al_2O_3 contents of the froth product. By contrast, the phosphate content of the froth and sink products remained relatively constant. Furthermore, the phosphate recovery (9.9%) was approximately equal to the solid recovery (9.8%). This finding suggests an entrainment mechanism for the fine phosphate present in these slimes.

By contrast, the MgO and CaO grades of the froth product were considerably higher than that in the feed. Similarly, the recoveries of the MgO (21%) and CaO (16%) were significantly higher than the solid recovery (9.8%) in the froth. These findings suggest a selective recovery of the coarser dolomite by flotation. As indicated above, our classification results revealed that MgO and CaO were concentrated in the coarse fractions. Thus, our flotation results are consistent with the classification studies.

Table 6. Composition of the Froth and Sink Products from Flotation of Phosphatic Slimes.

Parameter	Float		Sink	
	Grade	Recovery	Grade	Recovery
P ₂ O ₅ (wt. %)	7.17	10	7.07	90
MgO (wt. %)	9.96	21	4.24	79
CaO (wt. %)	21.1	16	12.5	84
SiO ₂ (wt. %)	17.3	6	31.5	94
Al ₂ O ₃ (wt. %)	9.7	10	15.6	90
Fe ₂ O ₃ (wt. %)	1.48	5	2.87	95
Solid Distribution (wt. %)		10		90

Two ml of Custofloat AR-27 (Westvaco) was added to 4 liters of Sample S-390. No dispersant was added in this case. After conditioning, the clay slimes were floated and the results obtained are shown in Table 7.

Table 7. Flotation Results Using Custofloat AR-27.

Parameter	Float		Sink	
	Grade	Recovery	Grade	Recovery
P ₂ O ₅ (wt. %)	8.01	11	7.50	89
MgO (wt. %)	5.05	13	4.17	87
CaO (wt. %)	14.8	12	12.8	88
SiO ₂ (wt. %)	29.9	10	33.5	90
Al ₂ O ₃ (wt. %)	15.4	10	17.4	90
Fe ₂ O ₃ (wt. %)	2.84	10	3.01	90
Solid Distribution (wt. %)		11		89

The results obtained with Custofloat AR-27 exhibit the same trends as those reported in Table 6 above and this will not be discussed further. Several other experiments were also carried out with using different concentrations of oleic acid, sodium silicate, Custofloat AR-27, and CA-99. In all of these tests, the concentration levels observed with the unclassified feed were modest.

CONTINUOUS FLOTATION EXPERIMENTS

In order to confirm the trends observed with the batch tests, continuous flotation experiments were carried out on the unclassified material. Table 8 shows the variation of particle size distribution with the different stages of feed condition. For this test, the unclassified feed was conditioned at pH of 9.2. The results confirm the trends obtained during the batch tests, i.e., addition of collector results in flocculation and a slight increase in the coarse fraction. For this particular case, it does appear that dodecane addition also produced a slight increase in the particle size distribution. Similar results have also been obtained with fine coal cleaning.

Table 8. Variation of Particle Size Distribution with Sample Treatment.*

Parameter	Sample ID		
	Feed	After Oleate	After Dodecane
$d_{10}, \mu\text{m}$	1.1	1.2	1.3
$d_{50}, \mu\text{m}$	4.9	5.1	5.2
$d_{90}, \mu\text{m}$	18.0	18.0	18.7

* 100 liters of slurry conditioned with 15 ml oleic acid followed by 10 ml of dodecane at pH of 9.2

The variation of froth product grade with flotation time is shown in Figure 8. In general, the results show that the froth grade remained fairly steady over the entire duration of the experiment. We have previously shown with coal flotation tests that steady state is achieved in a relatively short (about 2-3 minutes) with the use of the procedure outlined in this study (Ityokumbul and Trubelja 1998). Since the product grade did not vary appreciably with flotation time, we computed the average values for the entire flotation time and this is compared with the feed grade in Table 9. The results of these testing have confirmed the trends observed with the batch test work. As an example, the MgO and CaO contents of the froth product were increased, thus confirming the preferential concentration of dolomite during flotation. The slight lowering in the yield is attributed in part to the use of wash water in the continuous flotation tests. It is also noted that no sodium silicate was used to effect dispersion of the material.

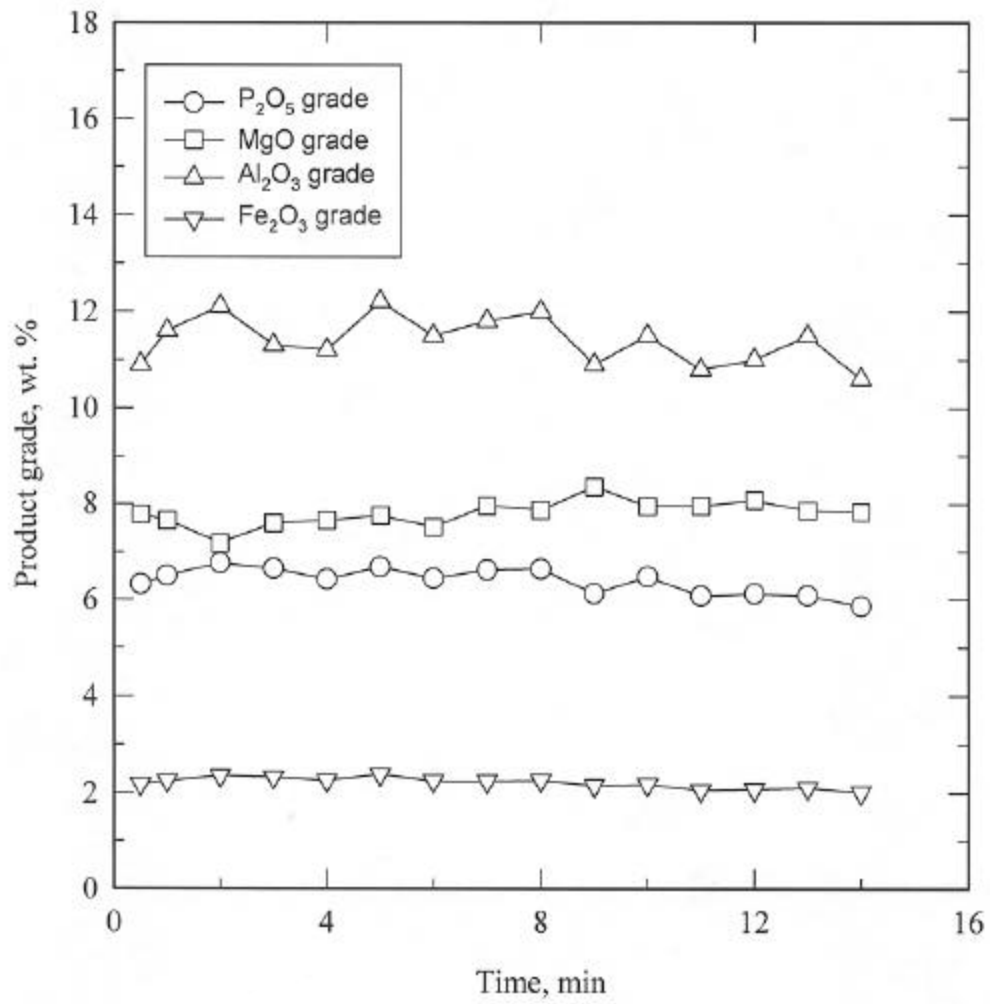


Figure 8. Variation of Product Grade with Flotation Time.

Table 9. Separation Results From Continuous Column Flotation Testwork.

Parameter	Grade			Distribution	
	Feed	Float	Sink*	Float	Sink
Solid Distribution (wt. %)				7	93
P ₂ O ₅ (wt. %)	8.33	6.37	7.50	5	95
MgO (wt. %)	4.63	7.8	4.17	12	88
CaO (wt. %)	14.2	14.8	12.8	7	93
SiO ₂ (wt. %)	31.0	26.3	33.5	6	94
Al ₂ O ₃ (wt. %)	16.0	11.4	17.4	5	95
Fe ₂ O ₃ (wt. %)	2.81	2.20	3.01	5	95

* Computed composition of the sink material.

EFFECT OF GAS VELOCITY

The variation of the concentrate flow rate, P₂O₅ and weight recoveries, and froth product grade with gas velocity is shown in Table 10. In general, the concentrate flow rate increases with gas velocity. However, in all these tests, the solid and phosphate mineral recoveries were almost identical. As indicated earlier, this suggests that the predominant mechanism for mineral recovery is entrainment. It is nevertheless noteworthy that the flotation results show an increase in the concentrate flow rate with gas velocity. For example, flotation has been recognized as being an interfacial phenomenon. Thus, the concentrate flow rate is expected to increase with increasing bubble surface area. Since the available bubble surface area for particle collection increases with gas velocity (Ityokumbul and Trubelja 1998), this observation is indeed a reasonable one.

Table 10. Effect of Air Flow Rate in Phosphate Recovery and Frother Product Grade.

Parameter	Air Flow Rate, Liters per Minute			
	1.5	2.5	3.5	4.5
P ₂ O ₅ recovery, %	7.1	17.3	20.1	24.1
Wt. Recovery, %	7.6	17.6	20.6	23.8
Concentrate flow rate, g/min Grade	45.0	106	141	155
P ₂ O ₅ (wt. %)	7.1	7.65	7.16	7.45
MgO (wt. %)	6.23	5.92	5.53	5.89
CaO (wt. %)	16.9	16.9	16.0	16.7
SiO ₂ (wt. %)	29.5	31.9	30.0	31.3
Al ₂ O ₃ (wt. %)	13.3	14.8	14.0	14.4
Fe ₂ O ₃ (wt. %)	2.92	3.05	2.91	3.02

EFFECT OF COLLECTOR DOSAGE

Figure 9 shows the effect of collector dosage on the concentrate flow rate. In general, the concentrate flow rate decreases with collector dosage, with the effect being more pronounced at higher airflow rates. In all the tests, only the MgO and CaO contents of the froth product were increased. This suggests that increasing the collector dosage promoted the recovery of the coarse dolomite in these slimes.

As indicated above, the concentrate flow rate decreased with collector dosage. This finding is significant. For example, the results show that in column flotation where deep froths are often employed to effect gangue rejection, increasing the collector concentration may produce froths with decreasing mobilities. It is therefore important that the selection of the collector and frother dosages for column flotation applications be done after careful study.

EFFECT OF DISPERSANT

The effects of sodium silicate addition on the concentrate flow rate, and product grade were studied and the results of these tests are shown in Table 11. In general, the addition of sodium silicate dispersant resulted in a lowering of the concentrate flow rate. For example, addition of 10 PPM of sodium silicate to the slurry reduced the concentrate flow rate from 99 g/min to 36 g/min at an air flow rate of 3.5 LPM. Increasing the airflow rate from 3.5 LPM to 4.5 LPM resulted in a modest increase in the concentrate flow rate. For all the continuous flotation tests, the concentrate grade was lower than that of the feed, thus suggesting that the fine phosphate mineral value was not amenable to concentration by flotation.

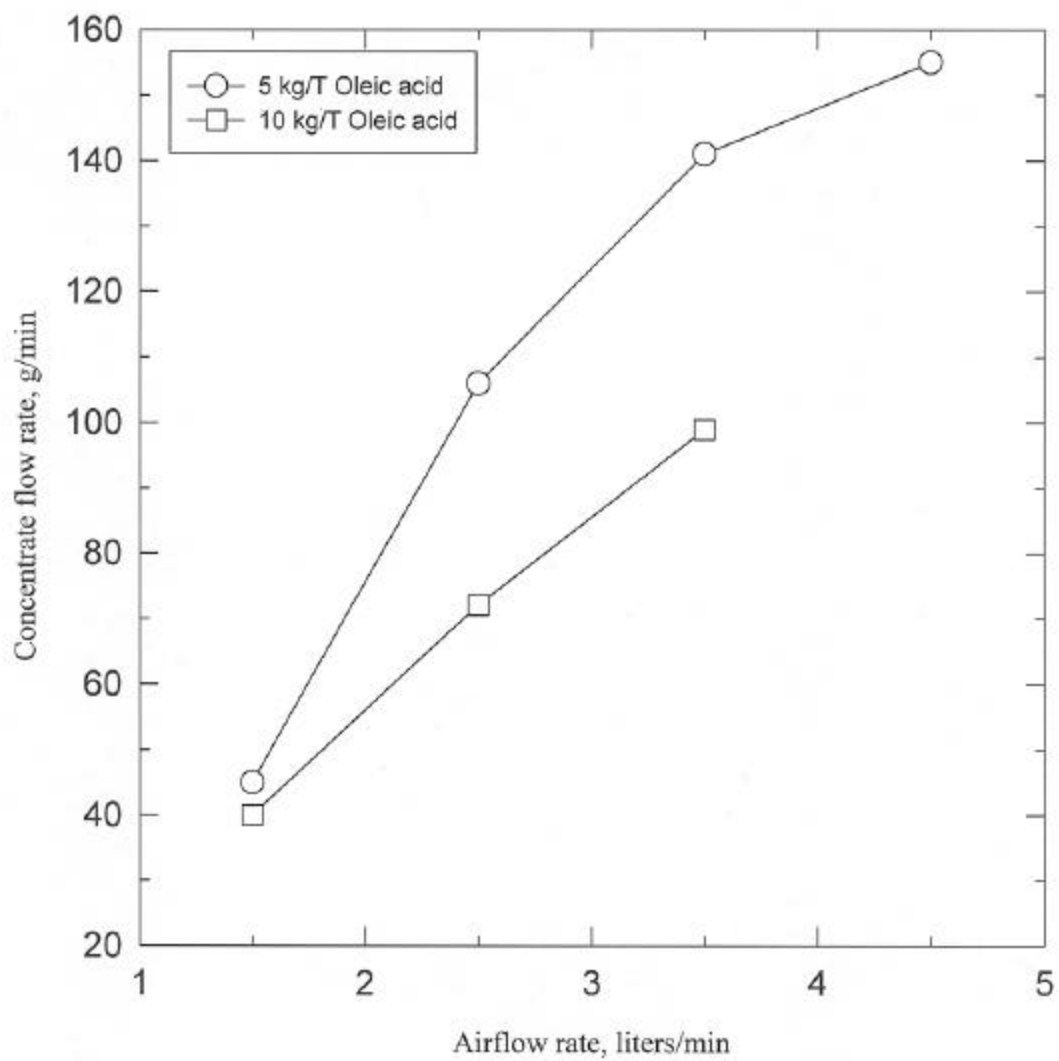


Figure 9. Effect of Collector Dosage on Concentrate Flow Rate.

Table 11. Effect of Dispersant in Product Yield and Grade.

Parameter	3.5 LPM	4.5 LPM
P ₂ O ₅ recovery, %	5.0	
Wt. Recovery, %	5.73	
Concentrate flow rate, g/min Grade	3.6	4.0
P ₂ O ₅ (wt. %)	6.59	6.34
MgO (wt. %)	6.72	7.02
CaO (wt. %)	17.2	17.8
SiO ₂ (wt. %)	32.0	30.0
Al ₂ O ₃ (wt. %)	13.3	12.2
Fe ₂ O ₃ (wt. %)	2.65	2.65

OVERALL COMPARISON

As indicated previously, it appears that the predominant recovery mechanism for mineral particles by flotation is entrainment. Figure 10 shows a parity plot for mineral component and solid recoveries. As expected, most of the mineral species exhibit a linear dependency, thus confirming the entrainment mechanism. This observation suggests that economic recovery of the phosphate resource present in these clay slimes by flotation alone is not possible. Our results are consistent with those reported earlier by Jordan et al. (1982). For example, Jordan et al. (1982) showed that for the finer phosphatic clay fraction, the product grade was even lower than the feed grade. Similar results have been obtained in this study with tailing slime samples from the IMC plant. Our feed was predominantly fine-sized material. to produce a 30% concentrate, results by Jordan et al suggest that a plus 20 micron feed is required. In the present study, this cut size is not feasible. Moreover, the P₂O₅ content of our sample was one-third that in their study.

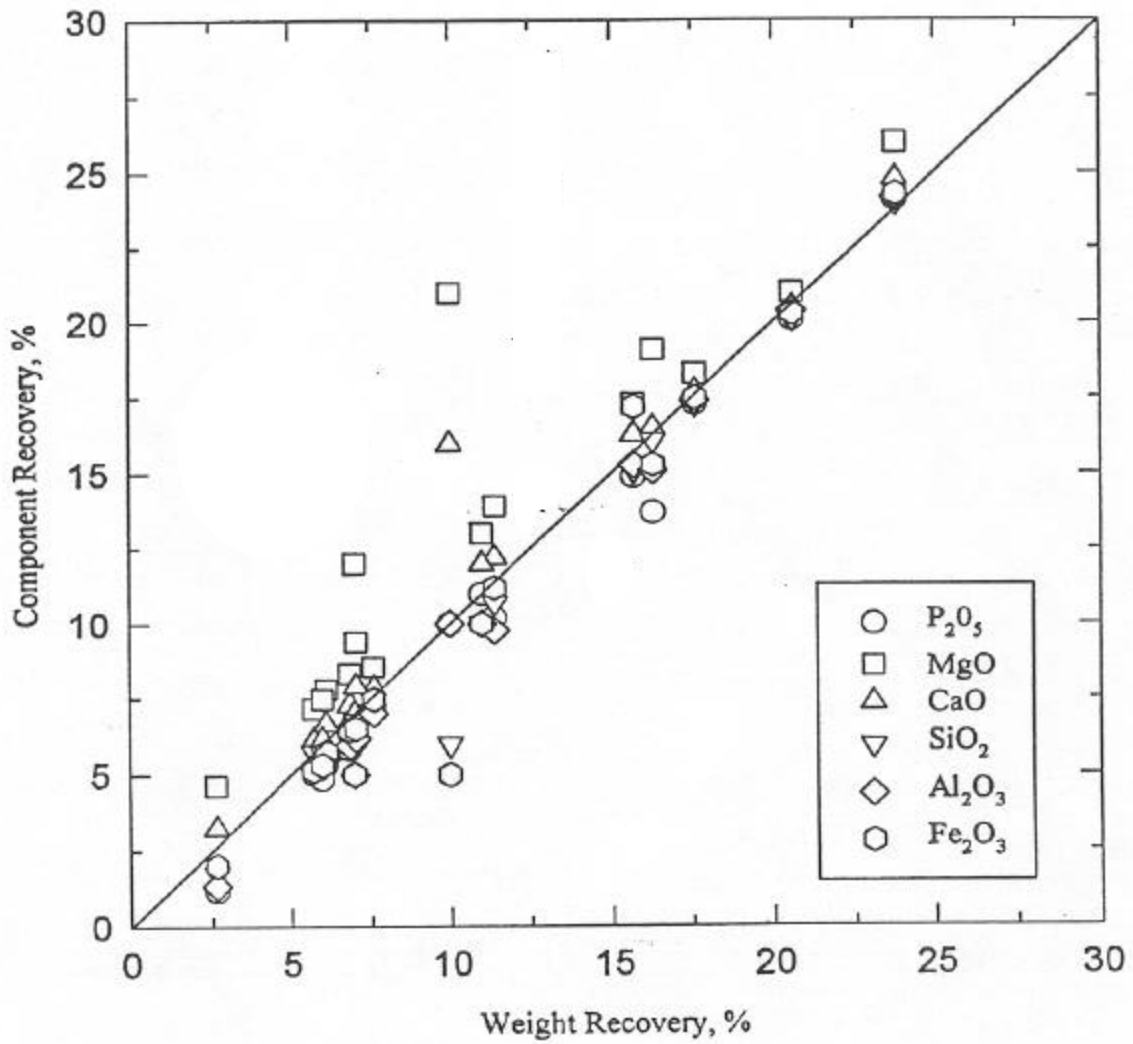


Figure 10. Overall Comparison of Flotation and Weight Percent Recoveries.

CONCLUSION AND RECOMMENDATIONS

Upgrading of the extremely fine phosphatic clay samples used in our study by flotation does not appear to be viable. However, it is conceivable that the use of another phosphatic clay slime sample with higher P_2O_5 content may yield different results. Unfortunately, the duration of the study did not permit this aspect of the study to be carried out. It is therefore recommended that other advanced separation processes be *additionally* evaluated for processing these clay slimes. Examples of processes that may be evaluated include:

- use of aqueous biphasic separation (Chaiko et al. 1993, McGaulley 1998)
- flocculation prior to flotation (Moudgil et al. 1995)
- solvent sublation (Smith and Valsaraj 1998).

The work reported here was carried out using phosphatic clay sample from one operating plant. Additional work on other phosphatic clay samples may also be carried out, as the particle size distribution of this sample was much finer than those reported previously in the literature. The extremely fine nature of the phosphatic clay sample suggests that the hydrocyclones at this IMC plant were very efficient with negligible misplacement of material.

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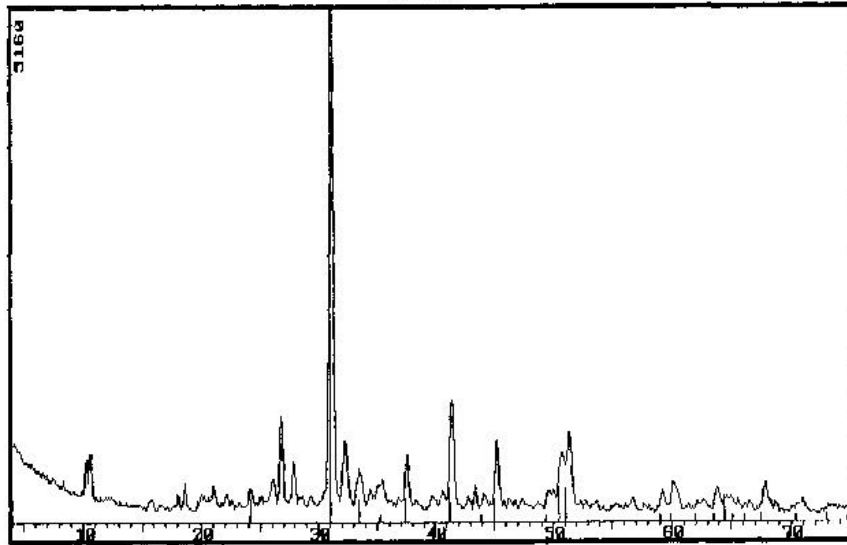
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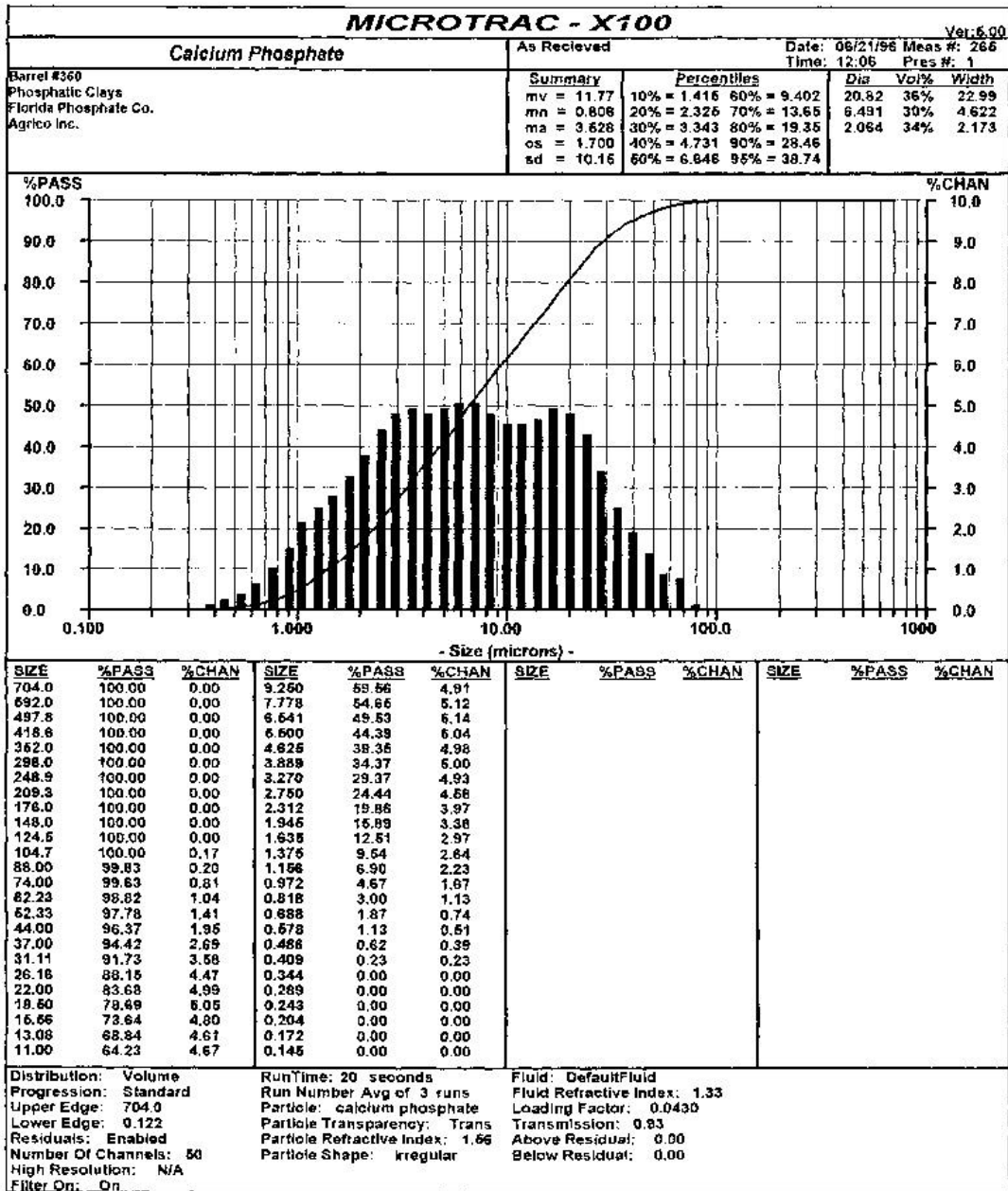
APPENDIX A

X-RAY DIFFRACTION PATTERN OF FLOTATION CONCENTRATE AND TYPICAL PARTICLE SIZE DISTRIBUTION OF PHOSPHATIC CLAY SLIMES

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Typical x-ray diffraction pattern for flotation concentrate. The lines represent the intensities for dolomite.



APPENDIX B
LITHIUM METABORATE FUSION TECHNIQUE

Dissolution of Silicates at Materials Characterization Lab

Lithium Metaborate Fusion Techniques

- Add *0.100 or 0.200 gm. of -100 mesh samples* to a pre-weighed vial containing *1.000 gm. of lithium metaborate*.
- Shake vials by hand, gently but thoroughly mixing the two components.
- Dump contents of vials into a graphite crucibles and insert crucibles into an oven preheated to 1000C.
- Pipette *100 ml of a 5% nitric acid* solution into watch glass covered Teflon beakers and add a stirring bar to each.
- After ten minutes at 1000C (allow a few minutes for oven to return to 1000C), place beakers on magnetic stirrers and start then swirl the contents of the crucibles to pick up any uncoalesced beads and dump into beakers.
- After a few minutes, carefully examine the graphite crucibles for any remaining melt, scrape off if present and add to beakers.
- Stir at least 15 minutes and transfer to polyethylene bottles for storage.

Tips and hints

- If melt sticks to graphite crucible and cannot be completely scraped out and dissolved, a smaller sample size will have to be used.
- Difficult samples such as zircons and high iron ores, will sometimes dissolve when fused in a 1:3 mix of sample to SiO_2 or Al_2O_3 . (e.g. .050 gm sample + 0.150 gm SiO_2 + 1.000 gm LiBO_2 .)
- This technique will not work for elements that are volatile at 1000C.
- Standards may be made by preparing a lithium metaborate matrix solution and using this to dilute down stock solutions.
- Standards may also be prepared by fusing known rock and/or ore standards (e.g. U.S.G.S. rock standards), along with the samples.
- Do not mix samples and standards prepared more than two weeks apart.
- Crucibles should be thoroughly wiped out with kimwipes in between samples.

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