# HIGH THROUGHPUT, HIGH SOLIDS FLOTATION OF 20 x 200 MESH NORTH CAROLINA PHOSPHATE IN A SHORT COLUMN CELL

by

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# **EXECUTIVE SUMMARY**

A pilot plant was constructed and operated to investigate the column flotation of 20 x 200 mesh North Carolina phosphate ore, using high throughput, high solids operating conditions developed at the NCSU Minerals Research Laboratory. Flotation was performed at 45% solids in a 9.5 cm (3.75 in.) diameter by 2.26 m (7.4 ft.) tall column at throughputs of 19.1  $t/hr/m^2$  (2.0 stph/ft²) and 31.7  $t/hr/m^2$  (3.3 stph/ft²).

Recoveries exceeding of 98% of the phosphate value were obtained at the lower throughput -- while obtaining concentrate

grades containing over 28%  $P_2O_5$  (61.2% BPL) at that recovery. Recoveries of about 95% of the phosphate value were obtained at the higher throughput, while making a concentrate averaging 27.6%  $P_2O_5$  (60.3% BPL). The concentrate mass loadings at the low and high throughputs were 19.5 and 32.0 g/min/cm², respectively.

In addition, a better understanding of the variables governing the column flotation of coarse nonmetallic minerals was gained. Low air flow rates adversely affected concentrate grade at the lower throughput. At the higher throughput, phosphate recovery decreased at an elevated air flow rate. An unexpected result of the study was the effect that frother type had upon phosphate metallurgy. Use of Nottingham Econofroth 925 provided better flotation selectivity than American Cyanamid F-65 at the low throughput. This is significant since the high throughput tests were conducted with F-65 exclusively. It is reasonable to expect further improvement in high throughput performance when the optimal frother type and dosage are determined.

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### **BACKGROUND**

Column flotation technology has steadily evolved and gained acceptance since Boutin and Tremblay (1964) patented the prototype Canadian column. In 1963, Hollingsworth (1967) filed the first U.S. patent claim for a countercurrent column. Although the purpose of this forerunner of the Flotaire column was to float coarse (+48 mesh) phosphate particles, column R & D since that time has overwhelmingly addressed fine particle applications. The feeds for these applications (i.e., ultrafine coal or metal-bearing sulfide ores) are typically 80% passing 74 microns -- and often much finer. Much of this fine particle work (e.g., Luttrell and

others (1988); McKay and others (1988); Parekh and others (1988); Ynchausti and others (1988)) has emphasized sparging systems and the importance of using a proper combination of operating variables, notably a net downflow of water (positive bias). In their reference book about the Canadian column, Finch and Dobby (1990) reviewed the definitions of column operating variables that have been adopted. Moreover, they reported the levels of variables used and the capacities obtained for a variety of fine sulfide mineral applications. The ranges were as follows:

- Bias Rate (cm/sec): 0.0 to +0.4
- Wash Water Rate (cm/sec): 0.2 to 0.5
- Froth Depth (m): 0.6 to 1.5
- Superficial Air Velocity (cm/sec): 0.8 to 3.0
- Carrying Capacity (g/min/cm<sup>2</sup>): 1.4 to 16.1

It has only been within the past two years that Soto and Barbery (1991) and Soto (1992) have reported the results of a comprehensive study about the column flotation of coarse particles. Their laboratory and pilot-scale sidestream test program was geared towards coarse (14 x 48 mesh) phosphate ore. Instead of using a deep froth with wash water, they injected elutriation water at the bottom of the cell to assist in levitating the fast-settling coarse particles. Detachment of bubbles from the coarse particles, which happens due to turbulence in mechanical cells, was further prevented by reducing the superficial air velocity. Excellent recoveries were obtained. The levels of variables used and capacity obtained were as follows:

- Bias Rate (cm/sec): -0.5 (i.e., net upflow of water)
- Wash Water Rate (cm/sec): none used
- Froth Depth (m): none
- Superficial Air Velocity (cm/sec): 0.8
- Column Throughput (t/hr/m²): 11.0 (at 96% P<sub>2</sub>O<sub>5</sub> recovery)

Soto (1992) also stated that the column throughput might be improved to  $15.1 \text{ t/hr/m}^2$  with some modification and optimization.

Because of the favorable reports regarding the efficiency and reduced costs of column flotation, the Minerals Research Laboratory (MRL) initiated an intensive column flotation test program in March 1991. Its purpose is to determine the feasibility of using column flotation to recover the many nonmetallic minerals produced in the state of North Carolina. Most of the flotation feeds treated are in the 20 x 200 mesh size range. This size range follows from at least one of the following two factors: 1) the mineralogical profiles of the ores and 2) product end-use specifications.

#### **OBJECTIVE**

The purpose of this study was to investigate the column flotation of 20 x 200 mesh North Carolina phosphate ore in a small pilot plant. The test objectives were to: 1) maximize throughput and minimize water consumption, 2) obtain the best possible metallurgical results, and 3) gain a better understanding of the relationship of column operating variables at high throughput and high solids.

#### SAMPLE

A 10 t bulk sample of flotation feed was received from Texasgulf, Inc. in Aurora, N.C and designated as MRL Laboratory No. 5886. The material was dried in batches at 110° C. and scalped at 20 mesh so that accurate feeding was possible in the pilot plant. This treatment resulted in the rejection of less than 2% of the material.

#### **PROCEDURES**

#### Reagents

Nottingham 925 Econofroth (EF 925), a water-soluble polyglycol ether, was added as a dilute solution to aid in bubble generation

during a portion of this study. However, this frother had a tendency to plug and foul the pilot plant flowmeters and sparger. Consequently, its use was discontinued in favor of American Cyanamid Frother 65 (F-65), a pure polyglycol.

Texasgulf, Inc. provided five gallon samples of the fatty acid/tall oil collectors used in its mill. These were Union Camp Unitol CTF (referred to hereafter as UC) and Nottingham 17-9761 (referred to hereafter as 17-9761). UC was used for the majority of tests. For flotation tests, the collector was partially saponified and diluted to the appropriate solution strength. No. 2 fuel oil was used as an extender during conditioning. The fuel oil/fatty acid ratio was maintained at 0.6 for all tests.

#### Pilot Scale Column

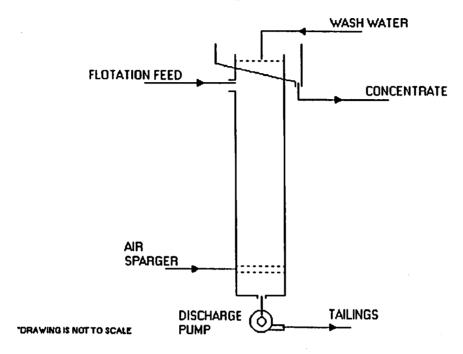
A 9.5 cm (3.75 in.) diameter pilot scale column (Figure 1) was constructed out of flanged sections of clear acrylic. This permitted the column height to be adjusted. For the phosphate test work, a total column height of 409 cm (13.4 ft.) was originally used. However, as experience was gained, it became obvious that a far shorter column was sufficient. Consequently, two 92 cm (two 3 ft.) sections were removed, resulting in a total height of 226 cm (7.4 ft.). Of this total height, 159 cm (5.2 ft.) comprised the collection zone and 67 cm (2.2 ft.) incorporated the concentration zone. The air sparger, located a distance of 25 cm from the column bottom, consisted of a porous polyethylene tube (20 micron mean pore size) that was obtained from the Deister Concentrator Company.

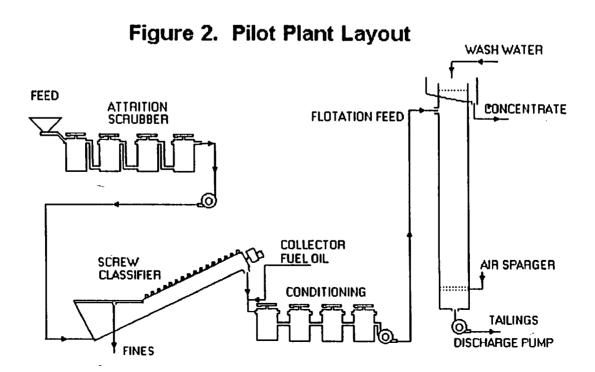
# Auxiliary Pilot Equipment

Proper preparation of flotation feed was critical to success. The auxiliary equipment (Figure 2) was configured to accurately control feed rate of ore, additions of water, flow rates of reagents, mixer tip speed, and mixer retention time.

Ore was fed dry by means of a Vibra-Screw volumetric feeder to a six-pot train of pilot-scale Wemco attrition scrubbers. A retention







time of approximately 4 minutes was used for each test.

The scrubber discharge was diluted and pumped to a 15 cm (6 in.) diameter Denver screw classifier. This screw washed the slurry and overflowed material finer than about 325 mesh. In addition, the underflow solids were dewatered to about 75% solids. (Because the feed was previously deslimed at Texasgulf, the amount of solids overflowing the screw was only about 0.5% of the feed.)

Sufficient water was added to the underflow solids to produce a pulp of approximately 65% solids for reagent conditioning. Conditioning with the fatty acid and fuel oil was performed in a four-pot train of cylindrical mixing vessels. The reagents were metered by means of peristaltic pumps. The pulp level in the conditioners was varied according to the ore feed rate to obtain a retention time of approximately 5 minutes. The conditioner discharged into a sump, which was pumped to the column feed inlet. Water was added as required to control the percent solids of the feed.

# Column Pilot Tests

Several series of tests were conducted (Table 1). The primary purpose was to maximize column capacity while obtaining the best possible metallurgy. A secondary purpose was to gain a better understanding of variables such as air flow rate, bias rate, and feed rate. As the test program evolved, the effect of frother on metallurgy became too profound to ignore; so, it was given some emphasis as well. The objectives each test series are listed in Table 1.

The range in levels of the variables used is shown in Table 2. Because the specific levels of the column variables used are proprietary, they are given in relative terms only. However, it can be said that these conditions are unique in that they differ from those reported by either Dobby and Finch (1990) or Soto (1992).

TABLE 1. OBJECTIVES OF PILOT PLANT TEST SERIES

OBJECTIVE
Mechanical shakedown of pilot plant.
Exploratory test run.
Tested column sensitivity to UC collector dosage using a high bias rate, a high air flow rate, and EF 925 frother.
Tested column response to air flow rate and bias rate using F-65 frother.
Repeated Series 3B using a lower UC collector dosage to improve concentrate grade.
Repeated Series 4 using a lower UC collector dosage and with a lower F-65 frother dosage to improve concentrate grade.
Repeated Series 5 at a higher feed rate. Obtained grab samples of concentrate and tail at various collector dosages.
Investigated the effect of frother type and frother dosage using 17-9761 collector.

TABLE 2. VARIABLE LEVELS USED IN COLUMN PILOT PLANT STUDY

VARIABLE	LEVELS	TEST SERIES
Feed Rate:	136 kg/hr (300 lbs./hr) 227 kg/hr (500 lbs./hr)	3A to 5, 7
Column Capacity:	19.5 t/hr/m <sup>2</sup> (2.0 stph/ft <sup>2</sup> ) 32.1 t/hr/m <sup>2</sup> (3.3 stph/ft <sup>2</sup> )	3A to 5, 7
Percent Solids	45% (in column)	3A to 7
Bias Rate: (negative)	Low/High Low/High	3A to 5, 7
Superficial Air Velocity:	Low/High Low/High	3A to 5, 7
Collector Type:	Union Camp (UC) Nottingham (17-9761)	3A to 6,
Collector Dosage: (tall oil)	0.15 kg/t (0.30 lb./ton) 0.16 kg/t (0.32 lb./ton) 0.18 kg/t (0.36 lb./ton) 0.20 kg/t (0.40 lb./ton)	5 6 3A, 4, 7 3B
Frother Type:	Nottingham (EF 925) American Cyanamid F-65	3A, 7 3B to 6
Frother Conc.	0.022 kg/t (0.044 lb./ton) 0.011 kg/t (0.022 lb./ton)" 0.0027 kg/t (0.0054 lb./ton)"	3A to 4, 7 5, 6 7

 <sup>&</sup>quot;Low" and "High" are relative terms. The "High" values used for tests at 136 kg/hr were the "Low" values used at 227 kg/hr.

<sup>→ -</sup> F-65 only.

#### RESULTS

# Sample Characterization

The chemical and size analysis of the feed sample are shown in Table 3.

# Column Test Series 3A

The metallurgical results are listed in Table 4. A good concentrate was obtained at a high recovery. The only negative result was the fact that the planned test series could not be completed, because the sparger was partially plugged by scale that precipitated out of the dilute frother solution.

# Column Test Series 3B

The metallurgical results are listed in Table 5. The sparger operated smoothly with the F-65 frother. The  $P_2O_5$  recoveries obtained were excellent. The data suggests that the concentrate grade was adversely affected when the lower air rate was used. In general, the concentrate grades obtained were not nearly as good as those obtained in Test Series 3A. This was owing to the increase in collector dosage and the frother used.

#### Column Test Series 4

Table 6 shows the metallurgical results of this test series. The UC collector dosage was reduced to the same level as in Test Series 3A. Although the  $P_2O_5$  recoveries were still good (> 95%), they were still not as high as those obtained in Test Series 3A. Moreover, the concentrate grades obtained were not nearly as high, although they were an improvement over Test Series 3B. Once again, the lower air rate appeared to reduce concentrate grade.

#### Column Test Series 5

During this test series, both the Unitol collector dosage and the F-65 frother dosage were reduced substantially in the hope of

TABLE 3. ANALYSIS OF PILOT PLANT FEED SAMPLE

Percent P<sub>2</sub>O<sub>5</sub> = 17.26 Percent Acid Insoluble = 43.46

			Scre	<u>een An</u>	<u>alysis</u>	<u>(Tyle</u>	r Mesh	)	
Individual							_150_		-200
% Retained	0.3	1.5	2.9	8.8	32.4	36.9	13.7	2.1	1.4

TABLE 4. SUMMARY OF RESULTS FOR TEST SERIES 3A

AIR	FLOW	RATE
****	1 1011	14211

	"LOW"			"HIGH"			
BIAS RATE	Det.	% P205 Recov.	Conc. P205	Assay BPL	% P205 Recov.	Conc. P205	Assay BPL
"LOW"	#1 #2						
"HIGH"	#1				98.4 98.3	28.29 28.98	61.84 63.34

Test Series was terminated before all conditions were tested due to a partially plugged sparger.

Feed Rate:

136 kg/hr (300 lbs./hr)

Throughput: Reagents:

19.1 t/hr/m<sup>2</sup> (1.96 stph/ft<sup>2</sup>) Collector = 0.18 kg/t (0.36 lbs./ton) UC.

Frother = 0.022 kg/t(0.044 lbs./ton) EF 925.

Grab Samples' at various Dosage % Recovery from assays & P<sub>2</sub>O<sub>5</sub> collector dosages: (kg/t) % BPL 0.16 94.6 28.41 62.09 0.19 97.3 28.40 62.08

<sup>· --</sup> Samples taken under high air rate, high bias rate conditions.

TABLE 5. SUMMARY OF RESULTS FOR TEST SERIES 3B

# AIR FLOW RATE

		"LOW"			1		
BIAS	Det.	% P2O5	Conc.	Assay	% P2O5	Conc.	Assay
RATE		Recov.	P2O5	BPL	Recov.	P2O5	BPL
"LOW"	#1	98.7	25.20	55.08	98.9	27.15	59.24
	#2	98.5	25.20	55.08	98.3	26.80	58.58
"HIGH"	#1	98.9	24.97	54.57	98.7	27.03	59.08
	#2	98.6	25.20	55.08	98.3	26.35	57.60

Feed Rate:

136 kg/hr (300 lbs./hr)

Throughput:

19.1 t/hr/m<sup>2</sup> (1.96 stph/ft<sup>2</sup>)

Reagents:

Collector = 0.19 kg/t (0.38 lbs./ton) UC. Frother = 0.022 kg/t (0.044 lbs./ton) F-65.

TABLE 6. SUMMARY OF RESULTS FOR TEST SERIES 4

#### AIR FLOW RATE

	"LOW"				"HIGH"		
BIAS	Det.	% P205	Conc.	Assay	% P2O5	Conc.	Assay
RATE		Recov.	P2O5	BPL	Recov.	P2O5	BPL
"LOW"	#1	95.6	25.89	56.59	95.6	27.03	59.08
	#2	95.6	26.80	58.58	95.1	26.92	58.84
"HIGH"	#1	94.7	26.58	58.10	94.1	26.92	58.84
	#2	96.1	25.77	56.33	95.3	26.23	57.33

Feed Rate: Throughput:

136 kg/hr (300 lbs./hr) 19.1 t/hr/m<sup>2</sup> (1.96 stph/ft<sup>2</sup>)

Reagents:

Collector = 0.17 kg/t (0.34 lbs./ton) UC. Frother = 0.022 kg/t (0.044 lbs./ton) F-65. making as clean a concentrate as that obtained in Test Series 3A. The results (Table 7) show that this idea was not successful. Product grades were slightly better than those obtained in Test Series 4, but the  $P_2O_5$  recovery tailed off considerably. Once again, the lower air rate appeared to cause a decrease in concentrate grade.

#### Column Test Series 6

Table 8 shows the results of this test series when the feed rate was increased from 136 kg/hr (300 lbs./hr) to 227 kg/hr (500 lbs./hr). The UC collector dosage was increased slightly from that used in Test Series 5. The frother dosage was the same, or half that used in Series 3B and 4. Good recoveries (about 92%-95%) were obtained when the lower air rate was used. As the reagent dosage data shows, recovery would have been higher if additional collector had been used. The recovery suffered at the higher air rate. Visual observation suggested that this was due to an increase in turbulence in the column. In general, the concentrate grades were better than those obtained in Test Series 3B, 4, or 5. However, they were still not as clean as those obtained in Test Series 3A.

# Column Test Series 7

The results of this Test Series, which considered frother type and dosage, are shown in Table 9. Collector 17-9761 was used instead of UC. Reducing the F-65 dosage to approximately 10% of the level used in Test Series 3B and 4 resulted in improved concentrate grades without adversely affecting recovery. Generation of bubbles was not a problem at this relatively low (0.0027 kg/t -- 0.0054 lb./ton) dosage of frother used. The test in which EF 925 was used as a frother resulted in improved recovery and grade.

TABLE 7. SUMMARY OF RESULTS FOR TEST SERIES 5

#### AIR FLOW RATE

		"LOW"			"HIGH"		
BIAS	Det.	% P2O5	Conc.	Assay	% P2O5	Conc.	Assay
RATE	No.	Recov.	P2O5	BPL	Recov.	P2O5	BPL
"LOW"	#1 #2	90.0	26.92 26.69	58.84 58.34	89.7 85.3	28.18 27.26	61.60 58.58
"HIGH"	#1	89.0	27.03	59.08	91.0	27.38	59.85
	#2	91.3	26.23	57.33	89.8	27.15	59.34

Feed Rate:

136 kg/hr (300 lbs./hr)

Throughput:  $19.1 \text{ t/hr/m}^2 (1.96 \text{ stph/ft}^2)$ 

Reagents:

Collector = 0.15 kg/t (0.30 lbs./ton) UC. Frother = 0.011 kg/t (0.022 lbs./ton) F-65.

TABLE 8. SUMMARY OF RESULTS FOR TEST SERIES 6

#### AIR FLOW RATE

	"LOW"				"HIGH"		
BIAS	Det.	% P2O5	Conc.	Assay	% P2O5	Conc.	Assay
RATE		Recov.	P2O5	BPL	Recov.	P2O5	BPL
"LOW"	#1	94.5	28.06	61.33	84.7	28.75	62.84
	#2	95.2	27.15	59.34	88.1	28.29	61.84
"HIGH"	#1 #2	94.5 91.2	27.26 27.49	59.58 60.09	90.4	28.98 28.18	63.34 61.60

Feed Rate:

227 kg/hr (500 lbs./hr)

Throughput: 31.7 t/hr

31.7  $t/hr/m^2$  (3.25  $stph/ft^2$ )

Reagents: Collector = 0.16 kg/t (0.32 lbs./ton) UC. Frother = 0.011 kg/t (0.022 lbs./ton) F-65.

Grab Samples' at various collector dosages:	Dosage (kg/t)	% Recovery from assays	% P <sub>2</sub> O <sub>5</sub>	% BPL
	0.14	87.4	28.29	61.84
	0.16	94.3	27.49	60.09
	0.20	97.0	26.80	58.58

<sup>· --</sup> Samples taken under low air rate, low bias rate conditions.

TABLE 9. SUMMARY OF RESULTS FOR TEST SERIES 7

#### FROTHER TYPE/DOSAGE

		F-65: 0.0027 kg/t (0.0054 lb./ton)			EF 925: 0.022 kg/t (0.044 lb./ton)		
AIR FLOW RATE	Det.	% P205 Recov.	Conc. P205	Assay BPL	% P205 Recov.	Conc. P205	Assay BPL
"LOW"	#1 #2	95.4	26.92	58.84			
"HIGH"	#1	95.6 96.1	27.49 27.61	60.09 60.35	97.4	28.06	61.33

Feed Rate: 136 kg/hr (300 lbs./hr) Throughput: 19.1  $t/hr/m^2$  (1.96  $stph/ft^2$ ) "High"

Bias Rate:

Collector: 0.18 kg/t (0.36 lbs./ton) 17-9761.

#### **DISCUSSION**

Several observations can be made by further comparison of the Figures 3 through 6 show a Grade vs. Recovery plot of the data points from Test Series 3B, 4, 5, and 6, respectively. data points in which the higher air rate was used have been filled in solid. The points from each Test Series are clustered around a certain region of the Grade vs. Recovery plot, depending upon the collector dosage used. Inspection of the points for any of the test series indicates that a decrease in air rate resulted in a reduction in concentrate grade. The reason for this is not known. However, it might be related to the fact that the lower superficial air velocity reduced turbulence in the column. The slight increase in turbulence at the higher air rate may have caused the agglomerates of reagentized phosphate to shear and release entrapped ganque particles.

As mentioned previously, the high air rate used during Test Series 3B, 4, and 5 was actually the same as the low air rate used

Figure 3. Effect of Air Flow Rate
on Metallurgical Performance (Series 3B)
(Low Throughput -- 0.20 kg/t Collector)

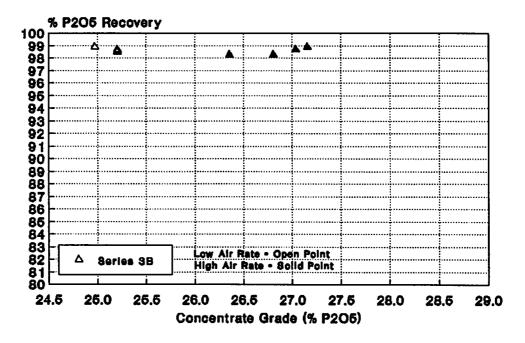


Figure 4. Effect of Air Flow Rate on Metallurgical Performance (Series 4) (Low Throughput -- 0.18 kg/t Collector)

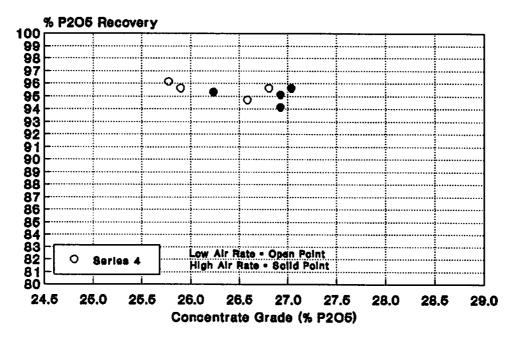


Figure 5. Effect of Air Flow Rate on Metallurgical Performance (Series 5) (Low Throughput — 0.15 kg/t Collector)

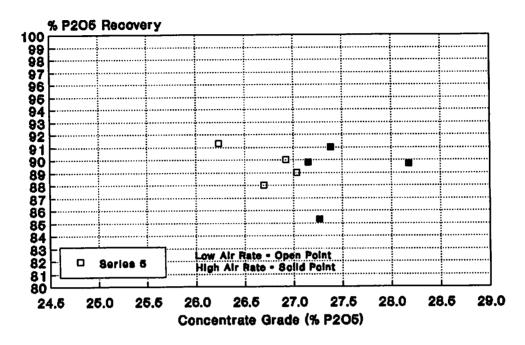
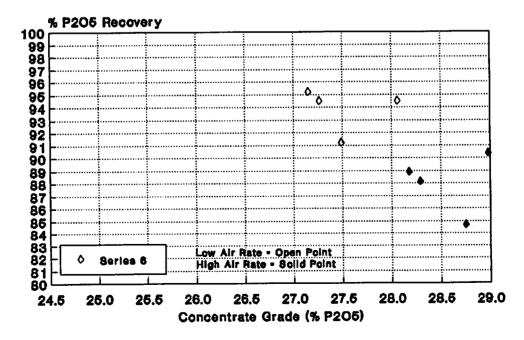


Figure 6. Effect of Air Flow Rate on Metallurgical Performance (Series 6) (High Throughput -- 0.16 kg/t Collector)



during Test Series 6, where column throughput was greatly increased. In addition, the high air rate used during Test Series 6 was considerably greater than the high air rates used in the other test series. Consequently, an increase in turbulence may have also been responsible for the drop in recovery that occurred when the high air rate was used during Test Series 6.

It should also be noted that the data from Tables 5-8 suggests that increasing the bias rate appeared to have caused slight reductions in concentrate grade. However, the reductions were much less than those caused by reduced air rate. In addition, there is scatter in the data. Consequently, more testing would be required to prove or disprove the effect of bias on grade.

Figure 7 shows a Grade vs. Recovery plot of the data points from Test Series 3A, 4, and 7. All of these test series used a collector dosage of 0.18 kg/t (0.36 lb./ton). Test Series 3A used 0.022 kg/t (0.044 lb./ton) of EF-925 with outstanding results. The same dosage of F-65 was used in Test Series 4, but mediocre concentrates were obtained. In Test Series 7, three tests were run using about 10% of the F-65 dosage used in Test Series 4. results were clearly better. Unfortunately, this comparison is compromised by the fact that different collectors were used in Series 4 (UC) and Series 7 (17-9761). However, Series 7 also included a single additional test run with 0.022 kg/t of EF-925, and the result was reasonably comparable to the excellent metallurgy obtained in Series 3A. All of this shows that the type of frother had a profound impact upon selective flotation. was considerably more active than the EF-925. Bubbles generated with it visually appeared to be finer than those generated with EF-925, and the flow regime appeared less turbulent. Thus, this frother may have caused a reduction in grade in much the same manner as a low air flow rate. Because the tests with the greatly reduced dosage of F-65 were performed with a different collector, it cannot be stated with certainty that this reduction improved metallurgy. However, the data does suggest that this improvement

Figure 7. Effect of Frother Type and Dosage on Metallurgical Performance (Low Throughput -- 0.18 kg/t Collector)

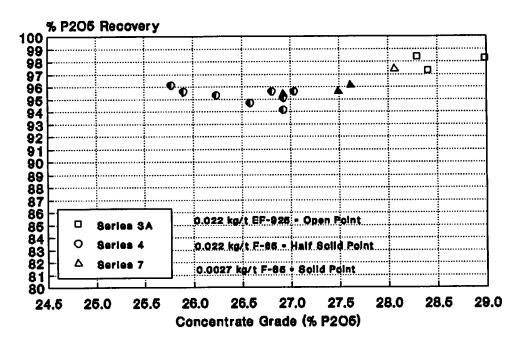
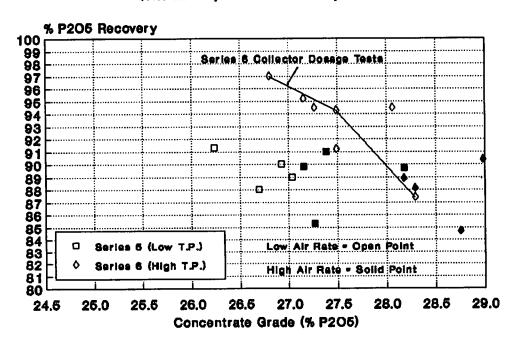


Figure 8. Effect of Column Throughput on Metallurgical Performance (19.1 t/hr/sq.m. vs. 31.8 t/hr/sq.m.)



occurred. Moreover, the minimum effective frother dosage has yet to be determined.

Figure 8 plots the data points of Test Series 5 (low throughput) together with the data points of Test Series 6 (high throughput). These sets of points are plotted together since the F-65 dosage was 0.011 kg/t (0.022 lb./t) for both test series. data from Test Series 6 also includes a partial Grade vs. Recovery curve for a range of UC collector dosages. The minimum dosage was 0.14 kg/t (0.28 lb./ton). A UC dosage of 0.15 kg/t (0.30 lb./ton) was used during Test Series 5. The UC dosage was 0.16 kg/t (0.32 lb./ton) during the steady-state tests of Test Series 6. This data suggests that the flotation at the higher throughput was more selective -- that is, the concentrate grades appear to be higher at an equivalent P,O, recovery. Whether this would have been the result if the more selective EF-925 frother had been used can only be answered through additional testing. However, it was certainly the result when a moderate dosage of F-65 was used. Again, the reason for this is not known. However, it could be related to the fact that the increase in superficial feed velocity, which was proportional to the 66.7% increase in feed rate, created more turbulence. This may have caused the agglomerates of reagentized phosphate minerals to shear and release entrapped gangue particles.

Nonetheless, the bottom line of Figure 8 is the fact that a flotation column cell can be made to perform well at a throughput of  $31.7 \text{ t/hr/m}^2$  (3.25 stph/ft<sup>2</sup>). In addition, the interaction of flotation chemistry and mixing with frother and turbulence must still be much better understood to optimize this high-throughput column.

# CONCLUSIONS

Table 10 lists the cause and effect relationships identified from this study. They are based upon the following conclusions:

TABLE 10. FACTORS AFFECTING RECOVERY AND GRADE

CAU	EFFECT		
FACTOR	TYPE OF CHANGE	GRADE	RECOVERY
Collector Dosage	Increase	-	+
	Decrease	+	-
Air Flow Rate (Low Throughput)	Increase	+	0
(Low Infoughput)	Decrease	-	0
Air Flow Rate (High Throughput)	Increase	+	_
(nigh infoughput)	Decrease	-	+
Frother Type	Use EF 925	++	0
	Use F-65		0
F-65 Dosage	Increase	-	0
	Large Decrease	+	0
Column Throughput	Increase	0	0
Bias Rate	Increase	0	0

<sup>+ =</sup> positive effect - = negative effect

<sup>++ =</sup> large positive effect -- = large negative effect

<sup>0 =</sup> no effect discernable at conditions tested

- At the lower throughput tested, a reduction in air flow rate did not affect recovery but reduced the concentrate grade.
- At the higher throughput tested, an increase in air flow rate caused a reduction in recovery; however, the concentrate grade was improved.
- Use of Nottingham Econofroth 925 frother afforded better metallurgical performance than American Cyanamid F-65 when used at a dosage of 0.022 kg/t (0.044 lb./ton) for the tests at the lower throughput. For a given recovery, the concentrate grade was substantially higher.
- Reducing the dosage of F-65 by half (i.e., to 0.0011 kg/t) did not seem to improve concentrate grade at the lower throughput tested.
- Reducing the dosage of F-65 substantially (i.e., to 0.0027 kg/t) seemed to improve concentrate grade without affecting phosphate recovery.
- The data from the low and high throughput tests with 0.011 kg/t F-65 frother suggested that selectivity was better at the higher throughput. However, the effect of throughput should be compared using more ideal frother conditions before drawing a conclusion with certainty.
- The effect of bias rate was not discernable at the ranges tested.

#### SUMMARY

Pilot-scale flotation tests were performed in a 2.26 m (7.4 ft.) high column cell. It was operated at 45% solids and throughputs of 19.1  $t/hr/m^2$  (2.0  $stph/ft^2$ ) and 31.7  $t/hr/m^2$  (3.3  $stph/ft^2$ ) with good metallurgical results. These were as follows:

- At a column throughput of 19.1 t/hr/m² (2.0 stph/ft²), recoveries of greater than 98% of the phosphate value were obtained with concentrate grades greater than 28% P₂O₅ (61.2% BPL). These metallurgical results were obtained when using Nottingham Econofroth 925 at the high air flow rate.
- At a column throughput of 31.7 t/hr/m² (3.3 stph/ft²), recoveries of approximately 95% of the phosphate value were obtained with an average concentrate grade of 27.6% P<sub>2</sub>O<sub>5</sub> (60.3% BPL). These metallurgical results were obtained when using American Cyanamid F-65 frother at the lower air flow rate tested.

The concentrate mass loadings at the low and high throughputs were 19.5 and 32.0 g/min/cm<sup>2</sup>, respectively.

The Nottingham frother was not tested at the higher throughput. It is possible that metallurgical performance at the high throughput would be improved with its use.

Successful scale-up and implementation of the high throughput and high solids operating conditions would offer advantages to plants processing relatively coarse feedstock. Plants using mechanical cells would realize reductions in energy costs, water consumption, and floorspace requirements. Plants using columns operated in a conventional manner could operate at a higher throughput and achieve an improvement in coarse particle recovery.

These high throughput, high solids conditions for operating a flotation column are far from optimized. Additional testing should be performed through in-plant sidestream tests to provide more representative feedstock for the column; then, a meaningful comparison with plant performance can be made. This testing should be conducted at progressively higher feed rates to identify the maximum capacity. A wide variety of frothers and frother dosages should also be tested.

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#### REFERENCES

- Boutin, P. and Tremblay, R.J. 1964. Method and Apparatus for the Separation and Recovery of Ores. Canadian Patent No. 694,547. Filed June 4, 1963.
- Finch, J.A. and Dobby, G.S. 1990. <u>Column Flotation</u>. Pergamon Press, Oxford, England; pps. 95 & 161.
- Hollingsworth, C.A. 1967. Fractionation-Flotation Process and Concentration of Minerals. U.S. Patent No. 3,298,519. Filed Oct. 23, 1963.
- Luttrell, G.H., Weber, A.T., Adel, G.T., and Yoon, R.H. 1988.
  Microbubble Flotation of Fine Coal. In <u>Column Flotation '88</u>.
  Society of Mining Engineers, Inc., Littleton, Colorado; pps. 205-212; Editor: K.V.S. Sastry.
- McKay, J.D., Foote, Jr., D.G., and Shirts, M.B. 1988. Column Flotation and Bubble Generation Studies at the Bureau of Mines. In Column Flotation '88. Society of Mining Engineers, Inc., Littleton, Colorado; pps. 173-186; Editor: K.V.S. Sastry.
- Parekh, B.K., Groppo, J.G., Stotts, W.F., and Bland, A.E. 1988.
  Recovery of Fine Coal from Preparation Plant Refuse Using Column Flotation. In Column Flotation '88. Society of Mining Engineers, Inc., Littleton, Colorado; pps. 227-234; Editor: K.V.S. Sastry.
- Soto, H. and Barbery, G. 1991. Flotation of Coarse Particles in a Counter-Current Column Cell. Minerals & Metallurgical Processing, Vol. 8, No. 1.; pps. 16-21; Editor: R.B. Bhappu.
- Soto, H. 1992. Development of Novel Flotation-Elutriation Method for Coarse Phosphate Beneficiation. Final Report. Laval University, Quebec, Canada. Prepared for the Florida Institute of Phosphate Research, Bartow, Florida.
- Ynchausti, R.A., McKay, J.D. and Foote, Jr., D.G. 1988. Column Flotation Parameters Their Effects. In Column Flotation <u>'88</u>. Society of Mining Engineers, Inc., Littleton, Colorado; pps 157-172; Editor: K.V.S. Sastry.