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PHOSPHATE ROCK TREATMENT FOR WASTE REDUCTION

Prepared by
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PHOSPHATE ROCK TREATMENT FOR WASTE REDUCTION

for

FLORIDA INSTITUTE OF PHOSPHATE RESEARCH

FIPR CONTRACT NO. 93-01-112R

Prepared by:

JACOBS ENGINEERING GROUP INC.

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Perspective

It has long been recognized that Florida phosphate rock can contain dolomite, either as relatively pure particles or as a mixture of cemented phosphate rock-dolomite particles. Techniques to remove the dolomite have not been overly successful unless the phosphate rock is finely ground and then subjected to beneficiation by flotation. Flotation has not been employed largely for two reasons: (1) the cost of grinding and (2) the problems associated with handling and shipping the finely ground material.

However when phosphate rock is used in the manufacture of phosphoric acid, it is ground before it can be reacted with sulfuric acid to form phosphoric acid. Since there is a no additional grinding cost for the ground phosphate rock available at this point, it would seem that this is an ideal point to attempt to remove the impurities such as dolomite.

Successful removal of the dolomite significantly reduces the sulfuric acid requirement by eliminating both $CaCO_3$ and $MgCO_3$, improves the acid quality by reducing the MgO content of the finished phosphoric acid, and should sharply reduce defoamer usage in the phosphoric acid reactor due to lower CO_2 content of the phosphoric acid plant feed rock.

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SECTION 1
SUMMARY

1.1 Background

Separation of calcite and/or dolomite from phosphate rock by flotation is becoming the practice in several phosphate mining areas where higher carbonate contamination is encountered. In almost all cases, the phosphate rock must be ground to liberate the calcite and/or dolomite so that it can be separated from the phosphate rock. However, with the level of carbonate contamination typically found in Florida phosphate rock, this type of processing scheme has not been adopted. This is due primarily to problems associated with grinding and handling the ground rock at the mine.

The investigation of carbonate flotation of the phosphate rock feed to the phosphoric acid plant as a means of reducing the calcium and magnesium content of the rock used to produce phosphoric acid is of interest because the rock to be treated is already ground for use in phosphoric acid manufacture. Reduction of calcium would reduce the quantity of phosphogypsum produced per ton of phosphoric acid the reduction of magnesium associated with the dolomitic limestone would improve acid quality. Under these conditions, the economics of flotation may prove to be attractive.

The Board of Directors of FIPR approved Jacobs proposal to evaluate the preliminary feasibility of carbonate flotation of phosphate rock feed as a means of reducing the calcium and magnesium content of the phosphoric acid feed. FIPR Contract No. 93-01-712R for bench scale testing was issued March 28, 1994.

The approved test program has six major elements or tasks that will be performed sequentially. The eight tasks as listed following.

Task Number	Task Description
1.0	Sample Collection
2.0	Characterization Studies
3.0	Flotation Process Evaluations
4.0	Flotation Process Optimization
5.0	Flowsheet Confirmation
6.0	Final Report

1.2 Test Materials

With the cooperation of local phosphate producers, three samples of flotation concentrate, three samples of washer pebble, three samples of ground reactor feed rock, and three samples of phosphoric acidic plant pond water were obtained for the study.

Commercially available flotation reagents categorized as collectors, depressants, modifiers and frothers were selected for evaluation.

Test materials and reagents used in the test program are described in Section 2.0.

1.3 Characterization Studies

The characterization studies examined three samples of plant ground phosphate rock (reactor feed), three samples of laboratory ground flotation concentrate, and three samples of laboratory ground pebble. In each case, chemical and mineralogical analyses were performed on selected size fractions of the ground rock.

Mineralogical testwork performed by FIPR identified the major mineral components as francolite, quartz, and dolomite. Minor amounts of calcite were also identified. Locked dolomite was not found in the -400 mesh fraction of any of the ground rock samples. The +400 mesh component of the ground phosphate rock, however, contained only minor amounts of carbonate minerals.

Sieve/chemical analyses data showed preferential grinding of the dolomite, but not the francolite. Preferential grinding of calcium carbonates was indicated only on ground rock samples with $CaO:P_2O_5$ ratios of 1.50 or greater.

Based on the characterization studies and initial tests on the flotation process evaluation task, a blend of pebble 3 and concentrate 1 was selected for the remaining testwork. This blend contained approximately 28% P_2O_5 and provided a flotation feed with sufficient free carbonates after grinding.

1.4 Flotation Process Evaluation

The purpose of the evaluation task was to select, on the basis of comparative laboratory test results a flotation process for removing carbonate gangue from phosphate rock. Accordingly, three inverse flotation processes were evaluated: the BRGM process, the BOM process, and the IMC process.

Results of the evaluation tests showed that the IMC process was superior to either the BRGM or BOM processes. Although most of the liberated carbonate minerals are concentrated in the -400 mesh fraction of the ground phosphate rock, all three processes evaluated were ineffective at treating only the -400 mesh material using a mechanical laboratory flotation cell. The +400 mesh component of the ground phosphate rock does not contain sufficient liberated carbonate minerals to warrant being processes separately.

1.5 Flotation Process Optimization

Eighty-six formal bench scale flotation tests were conducted on two phosphate rock samples to determine which parameters of the IMC process could be changed to improve the separation of carbonate minerals from phosphate rock. To achieve this objective, statistically designed tests were performed on ground pebble 3 and aground blend of pebble 3 and concentrate 1.

The optimization tests demonstrated improved performance for both test feeds; however, acceptable quality phosphate rock was not obtained from pebble 3 at satisfactory P_2O_5 recovery. Test results for the blended phosphate rock sample are given below.

	Feed Stock	Concentrate
% P_2O_5	27.66	28.95
% CaO	43.88	43.46
% MgO	1.29	0.62
% P_2O_5 Recovery	100.0	93.0
CaO:P_2O_5 Ratio	1.586	1.501

The optimization tests demonstrated that pond water is an acceptable substitute for H_2SO_4 for pH control during grinding, conditioning and flotation. Froth depth, flotation cell size and cell rotor rpm were also shown to influence flotation performance.

1.6 Flowsheet Confirmation

Flowsheet confirmation tests (Task 5.0) were conducted at the optimum levels of conditioning and flotation parameters identified from the process optimization test results (Task 4.0). The confirmation tests were performed on a ground blend of pebble 3 and concentrate 1 and consisted of locked cycle tests to examine the effect of tailings water and concentrate water recycle. The locked cycle tests demonstrated acceptable flotation performance; however, recycle water had a slight adverse effect on flotation selectivity. The use of flocculant to facilitate tailings dewatering also reduced flotation selectivity when recycle water was used in flotation.

A minerals and chemical component balance was developed from the confirmation tests results and the characterization test data given in Section 3. This balanced formed the basis for the PFD materials balance for a carbonate flotation module installed in a 1000 tpd P_2O_5 phosphoric acid plant.

1.7 Capital and Operating Costs

The capital cost of-a flotationmodule added to a 1000 ton P_2O_5 per day phosphoric acid plant was estimated based on in-house Jacobs cost ratios (factors) and a priced equipment list developed from the process flowsheet and materials balance given in Section 7.

The estimated capital cost to construct the flotation module is $5.15 million. The order-of-magnitude grade estimate (±25% accuracy) includes the materials and equipment, and the cost of engineering, procurement and construction. The estimate is based on present-day pricing with no forward escalation included.

The total direct operating costs for the flotation module are $1.7 million per year, equivalent to $1.35 per ton of product, or $4.78 per ton P_2O_5. Operating cost details are presented in Section 7.

1.8 Recommendations

The Phase I testwork demonstrated the technical feasibility of carbonate flotation of the phosphate rock feed to the phosphoric acid plant as a means of reducing the calcium and magnesium content of the rock used to produce phosphoric acid. Benefits of the flotation process also include:

- higher consumption of pond water
- reduction in phosphogypsum production
- lower cost to produce DAP.

Pond water used in the flotation process is equivalent to 0.07 tons per ton of P_2O_5 in the reactor feed. This equates to an approximate 30% increase in pond water consumption relative to the amount typically used in wet grinding of the phosphate rock.

The amount of phosphogypsum produced using concentrate from the carbonate flotation process is about 3% less than phosphogypsum produced with untreated feed. This amounts to a reduction of over 42,000 tons per year for a 1,000 tpd P_2O_5 phosphoric acid plant.

Using the Jacobs DAPCOST model, a $7.06 per ton DAP cost savings is projected. The cost projection is based on the rock characteristics and current prices for sulfur and ammonia. The major elements of savings consist of a 3% reduction in sulfuric acid consumption and a penalty charged to the untreated rock because the MER of the resultant filter acid it too high to produce 18-46-0 DAP.

Accordingly, a Phase II program is recommended to more precisely define the technical and economic benefits of utilizing flotation to facilitate reducing the carbonate content of phosphoric acid plant reactor feed. The recommended Phase II program includes:

- bench scale flotation testing of two different plant feeds using mechanical and column flotation cells,
- pilot scale flotation of additional feed using a column flotation cell,
- technical/economic analysis of the pilot plant test results and updating of the Phase I cost estimates.

The Phase II program will provide a comprehensive and more realistic basis for evaluating the potential of the flotation process for Florida phosphate rock.

SECTION 2
PROCEDURES AND TEST MATERIALS

2.1 Procedures

In order to obtain reproducible results and minimize error, the testwork presented in this report was conducted utilizing analytical procedures approved by the Association of Florida Phosphate Chemists and the ZW test procedures listed below by procedure number.

001.2 Feed Blending and Sampling
004.2 Reagent Preparation and Use
009.0 Rougher Flotation Bench Testing - BRGM Process
010.0 Rougher Flotation Bench Testing - IMC Process
011.0 Rougher Flotation Bench Testing - USBM Process

A step-by-step description of each of the above procedures is presented in Appendix A.

2.2 Test Samples

The objective of the study was to investigate the use of flotation technology to remove liberated calcium and/or magnesium carbonates from phosphate rock after grinding but before acidulation. Accordingly, flotation concentrate, washed pebble and ground phosphate rock were specified for the testwork.

Three pebble samples and three flotation concentrate samples were collected from phosphate mines as identified in Table 2-1. Three ground phosphate rock samples and three pond water samples were collected from the phosphoric acid plants also listed in Table 2-1.

At each sample collection site the personnel were exceedingly cooperative and helpful. At the mine sites, concentrate and pebble were placed in separate lined 55 gallon drums, then transported to the ZW laboratory where they were unloaded, blended and sampled in accordance with lab procedure 001.2, and in preparation for Task 2 - Characterization Studies.

Table 2-1

Test Samples*

Sample Identification	Quantity	Sample Source
Flotation concentrate 1	500 lbs	IMC/Agrico - Noralyn
Flotation concentrate 2	500 lbs	IMC/Agrico - Ft. Green
Flotation concentrate 3	500 lbs	IMC/Agrico - Four Corners
Pebble 1	1,000 lbs	Mobil - Big Four
Pebble 2	1,000 lbs	IMC/Agrico - Ft. Green
Pebble 3	1,000 lbs	IMC/Agrico - Four Corners
Reactor feed rock 1	40 lbs	Cargill - Bartow
Reactor feed rock 2	40 lbs	IMC/Agrico - New Wales
Reactor feed rock 3	40 lbs	IMC/Agrico - South Pierce
Pond water 1	45 gal	Cargill - Bartow
Pond water 2	45 gal	IMC/Agrico - New Wales
Pond water 3	45 gal	IMC/Agrico - South Pierce

* quality of pebble and concentrate obtained for this study is not necessarily the quality of the run-of-mine products from the listed sample sources

The ball mill discharge from each of the listed chemical plants was sampled to obtain the required ground phosphate rock. One 55 gallon drum of pond water was also collected from each chemical plant site. The pond water samples and the ground phosphate rock samples were then transported to the ZW laboratory where representative samples were obtained for carrying out Task 2.

2.3 Reagents

In Task 3 - Flotation Process Evaluation, three inverse flotation processes for removing carbonate gangue from phosphate rock were evaluated. The processes tested were the BRGM process, the IMC process and the BOM process. The chemical reagents used in the flotation test program are categorized as collectors, depressants, modifiers and frothers.

2.3.1 Collectors

The anionic collectors for carbonates used in the test program are listed below:

- Flotinor SM-15: phosphoric ester
  Hostaphat MDIT: phosphoric ester
  Hoechst Aktienoqesellschaft Marketing
  D-6230 Frankfurt am Main 80
  Germany

- Westvaco CCS-502: Sulfonated oleic acid
  Westvaco Corp.
  P.O. Box 237
  Mulberry, FL 33860

- Westvaco L-5: tall oil*
  Westvaco Corp.
  P.O. Box 237
  Mulberry, FL 33860

  * made up as a 10% emulsion

2.3.2 Depressants

The three phosphate depressants used in the test program are listed below:

- Westvaco CCD-2112
  Westvaco Corp.
  P.O. Box 237
  Mulberry, FL 33860

- Fluosilicic Acid
  Aldrich Chemical Corp., Inc.
  P.O. Box 355
  Milwaukee, Wis. 53201

- Sodium Tripolyphosphate (STP)
  Fisher Scientific
  7464 Chancellor Dr.
  Orlando, FL
  This reagent is more correctly called STPP; however, the less correct abbreviation STP has been used in this report.

- Sodium Hexafluorosilicate
  Aldrich Chemical Corp., Inc.
  P.O. Box 355
  Milwaukee, WI 53201

2.3.3 Modifier

Chemical reagents that modulate the action of collector reagents by changing the surface characteristics of minerals are called modifiers. A listing of the modifiers used in the test program follows:

- Sodium Hydroxide (NaOH)
  Fisher Scientific
  7464 Chancellor Dr.
  Orlando, FL

- Sulfuric Acid (H_2SO_4)
  Fisher Scientific
  7464 Chancellor Dr.
  Orlando, FL

2.3.4 Frothers

This group of reagents reduces the surface tension of water and promotes the formation of a stable froth. One reagent used for frothing was:

Dowfroth 250
Dow Chemical Co.
Midland, Michigan

2.4 Water

Lakeland tap water was used in all tests, except those noted for pond water and/or recycle water use. Laboratory tap water analyses are reported in Section 6 (Tables 6-1 and 6-4).

SECTION 3
CHARACTERIZATION STUDIES

3.1 Objective

The purpose of the characterization studies was to determine if size reduction liberates the minerals and if differential hardness segregates the minerals. Identification of any material or size fractions which do not require and/or do not benefit from carbonate flotation was also an objective.

3.2 Composition of Rock and Water Samples

Sieve and chemical analyses were performed on the three reactor feed samples (plant ground phosphate rock samples), the three laboratory ground concentrate samples, and the three laboratory ground pebble samples.

Laboratory ground samples were generated by taking representative samples from the three concentrate and pebble samples listed in Table 2-1 and grinding to approximately 50% passing 200 mesh using a bench rod mill. After milling, the ground pulp was wet screened at 400 mesh. Both fractions (+400 and -400 mesh) were oven dried and then the +400 mesh fraction was screened on the following sieves: 35, 48, 65, 100, 150, 200, 270 and 400 mesh. After weighing the individual sieve fractions, representative samples of each fraction were analyzed for the following chemical components: P_2O_5, insol, CaO, MgO, Fe_2O_3, and Al_2O_3. The sieve and chemical analysis results for concentrate samples 1, 2, and 3 are given in Table 3-1. Table 3-2 gives the same data for pebble samples 1, 2, and 3.

Sieve and chemical analysis of the three reactor feed samples were obtained in a similar manner. Each sample was first wet screened at 400 mesh and then oven dried. The +400 mesh fraction was screened on the following sieves: 14, 20, 28, 35, 48, 65, 100, 150, 200, 270 and 400 mesh; and then each sieve fraction was analyzed for P_2O_5, insol, CaO, MgO, Fe_2O_3, and Al_2O_3. The sieve and chemical analysis data for the three reactor feed samples are presented in Table 3-3(a), 3-3(b), and 3-3(c).

Table 3-1

Ground Concentrate Sieve and Chemical Analyses

	Chemical Analysis					Component Distribution					
FRACTION (Mesh)	P_2O_5	Insol	CaO	MgO	I&A	% Wt.	P_2O_5	Insol	CaO	MgO	I&A
Concentrate 1											
48 x 65	31.56	5.23	46.53	0.48	2.68	0.8	0.9	0.9	0.8	0.5	0.8
65 x 100	31.76	4.80	46.98	0.51	2.30	11.0	11.3	10.2	11.2	6.2	9.3
100 x 150	31.42	5.89	46.62	0.51	2.23	18.7	19.1	21.4	18.9	10.6	15.4
150 x 200	31.08	6.48	46.12	0.54	2.44	17.3	17.6	21.9	17.3	10.4	15.6
200 x 270	30.79	6.55	45.70	0.56	2.63	13.3	13.4	17.0	13.2	8.3	13.0
270 x 400	30.79	6.04	45.95	0.66	2.63	7.7	7.7	9.1	7.7	5.7	7.5
-400	29.48	3.21	45.61	1.68	3.33	31.2	30.0	19.5	30.9	58.3	38.4
Calculated Head	30.66	5.13	46.08	0.90	2.70	100.0	100.0	100.0	100.0	100.0	100.0
Concentrate 2											
48 x 65	32.68	4.04	47.53	0.39	2.17	0.5	0.5	0.4	0.5	0.4	0.4
65 x 100	32.43	3.81	47.70	0.40	2.06	6.7	6.7	6.1	6.8	6.2	5.9
100 x 150	32.24	4.67	47.20	0.41	2.10	18.7	18.7	20.8	18.8	17.7	16.7
150 x 200	32.05	5.03	46.95	0.41	2.17	20.0	19.9	23.9	20.0	18.9	18.4
200 x 270	31.90	5.55	46.30	0.41	2.18	14.8	14.6	19.5	14.5	13.9	13.6
270 x 400	32.00	5.03	46.45	0.41	2.18	7.9	7.9	9.5	7.9	7.5	7.4
-400	32.58	2.65	47.13	0.49	2.82	31.4	31.7	19.8	31.5	35.4	37.6
Calculated Head	32.25	4.21	46.97	0.43	2.36	100.0	100.0	100.0	100.0	100.0	100.0
Concentrate 3											
35 x 48	31.34	6.94	34.84	0.39	1.78	0.1	0.1	0.1	0.1	0.1	0.1
48 x 65	32.66	3.43	48.36	0.42	1.94	1.3	1.4	0.8	1.4	0.9	1.1
65 x 100	32.23	4.32	47.65	0.43	1.90	8.7	9.0	6.3	8.9	6.1	7.6
100 x 150	31.56	6.36	46.62	0.44	1.86	17.7	17.9	19.1	17.8	12.7	15.1
150 x 200	30.94	7.86	45.78	0.45	1.88	20.4	20.2	27.2	20.2	14.9	17.7
200 x 270	30.80	7.98	45.53	0.47	1.89	13.8	13.7	18.7	13.6	10.6	12.1
270 x 400	31.08	6.83	46.12	0.53	2.01	8.3	8.3	9.6	8.3	7.1	7.7
-400	30.82	3.60	46.21	0.98	2.81	29.7	29.4	18.2	29.7	47.6	38.6
Calculated Head	31.14	5.89	46.24	0.61	2.17	100.0	100.0	100.0	100.0	100.0	100.0

Table 3-2

Ground Pebble Sieve and Chemical Analyses

	Chemical Analysis					Component Distribution					
FRACTION (Mesh)	P_2O_5	Insol	CaO	MgO	I&A	% Wt.	P_2O_5	Insol	CaO	MgO	I&A
Pebble 1											
35 x 48	29.93	12.44	43.54	0.28	1.55	0.8	0.8	0.9	0.8	0.4	0.7
48 x 65	30.94	10.30	45.28	0.28	1.56	5.1	5.3	4.7	5.3	2.4	4.2
65 x 100	29.98	12.69	44.12	0.28	1.51	19.2	19.2	21.6	19.3	9.1	15.3
100 x 150	29.22	15.46	42.87	0.29	1.46	14.6	14.3	20.0	14.2	7.1	11.3
150 x 200	28.93	15.96	42.29	0.29	1.48	12.7	12.3	18.0	12.2	6.3	10.0
200 x 270	29.46	14.25	42.95	0.34	1.58	9.8	9.7	12.4	9.6	5.6	8.2
270 x 400	29.84	12.01	43.95	0.53	1.70	6.9	6.9	7.4	6.9	6.2	6.2
-400	30.35	5.43	44.92	1.20	2.69	30.9	31.5	15.0	31.7	62.9	44.1
Calculated Head	29.84	11.24	43.88	0.59	1.89	100.0	100.0	100.0	100.0	100.0	100.0
Pebble 2											
35 x 48	28.37	13.36	42.76	0.46	2.43	0.1	0.1	0.1	0.1	0.1	0.1
48 x 65	29.97	8.36	45.45	0.48	2.37	2.0	2.0	1.9	2.0	0.9	1.9
65 x 100	29.53	9.16	44.87	0.51	2.24	12.9	13.3	13.2	12.9	5.9	12.0
100 x 150	28.85	11.54	44.03	0.55	2.04	15.3	15.5	19.8	15.1	7.6	13.0
150 x 200	28.47	12.49	43.29	0.58	2.02	14.1	14.0	19.7	13.6	7.4	11.8
200 x 270	28.66	11.95	43.45	0.60	2.05	12.1	12.2	16.3	11.8	6.6	10.4
270 x 400	29.05	10.16	43.95	0.71	2.15	7.6	7.7	8.6	7.5	4.8	6.8
-400	28.01	5.07	46.06	2.06	2.95	35.9	35.2	20.4	37.0	66.7	44.0
Calculated Head	28.60	8.93	44.71	1.11	2.41	100.0	100.0	100.0	100.0	100.0	100.0
Pebble 3											
35 x 48	20.53	23.95	32.32	0.50	2.13	0.1	0.1	0.1	0.1	0.0	0.1
48 x 65	26.72	16.50	41.04	0.77	1.96	0.9	1.0	1.3	0.9	0.3	0.8
65 x 100	27.40	13.46	42.45	1.02	1.90	8.3	8.9	9.3	8.4	3.7	7.3
100 x 150	26.77	14.59	41.62	1.33	1.72	15.1	15.8	18.1	15.0	8.8	11.9
150 x 200	26.05	16.43	41.49	1.41	1.69	16.6	16.9	22.4	16.3	10.2	12.8
200 x 270	25.95	16.50	40.97	1.48	1.74	13.8	14.0	18.7	13.4	9.0	11.0
270 x 400	26.19	14.80	41.79	1.59	1.84	8.6	8.8	10.4	8.5	6.0	7.2
-400	24.10	6.57	43.07	3.87	2.92	36.6	34.5	19.7	37.4	62.0	48.9
Calculated Head	25.56	12.17	42.12	2.28	2.18	100.0	100.0	100.0	100.0	100.0	100.0

Table 3-3(a)

Reactor Feed 1 Sieve and Chemical Analyses

	Chemical Analysis					Component Distribution					
FRACTION (Mesh)	P_2O_5	Insol	CaO	MgO	I&A	% Wt.	P_2O_5	Insol	CaO	MgO	I&A
+14	30.94	7.29	45.19	0.39	2.46	0.2	0.2	0.2	0.2	0.2	0.2
14 x 20	30.03	9.50	44.27	0.39	2.38	0.3	0.3	0.3	0.3	0.3	0.3
20 x 28	28.74	13.40	42.77	0.37	2.21	0.5	0.5	0.7	0.5	0.4	0.4
28 x 35	29.17	12.56	42.85	0.37	2.24	1.9	1.8	2.6	1.8	1.8	1.8
35 x 48	30.42	9.22	44.86	0.40	2.20	5.5	5.5	5.6	5.5	5.5	5.2
48 x 65	30.85	7.90	45.11	0.40	2.23	14.0	14.1	12.2	14.2	13.9	13.3
65 x 100	30.65	8.74	44.77	0.40	2.23	20.8	21.0	20.1	20.9	20.7	19.9
100 x 150	29.60	11.93	43.10	0.36	2.20	10.0	9.8	13.2	9.7	9.0	9.5
150 x 200	29.17	13.01	42.52	0.36	2.18	8.9	8.5	12.8	8.5	8.0	8.3
200 x 270	29.41	11.91	43.10	0.37	2.25	7.5	7.2	9.8	7.3	6.9	7.2
270 x 400	30.32	9.91	44.52	0.39	2.26	5.3	5.3	5.8	5.3	5.2	5.1
-400	31.40	6.05	45.91	0.45	2.68	25.1	25.8	16.7	25.8	28.1	28.8
Calculated Head	30.47	9.07	44.56	0.40	2.34	100.0	100.0	100.0	100.0	100.0	100.0

Table 3-3(b)

Reactor Feed 2 Sieve and Chemical Analyses

	Chemical Analysis					Component Distribution					
FRACTION (Mesh) Reactor Feed 2	P_2O_5	Insol	CaO	MgO	I&A	% Wt.	P_2O_5	Insol	CaO	MgO	I&A
+14	30.08	9.70	45.12	0.49	1.84	0.1	0.1	0.1	0.1	0.1	0.1
14 x 20	28.50	12.54	43.29	0.60	1.94	0.5	0.5	0.6	0.5	0.5	0.5
20 x 28	28.74	12.44	43.45	0.60	1.92	1.9	1.9	2.2	1.9	1.7	1.8
28 x 35	28.98	11.68	43.62	0.59	1.94	8.9	8.8	9.7	8.8	7.9	8.3
35 x 48	29.27	11.47	44.20	0.55	1.90	12.8	12.8	13.7	12.8	10.5	11.6
48 x 65	29.46	11.44	44.28	0.51	1.87	14.9	14.9	15.8	14.8	11.3	13.3
65 x 100	29.50	11.71	44.12	0.49	1.91	16.0	16.1	17.5	15.9	11.7	14.7
100 x 150	28.88	13.17	43.04	0.48	2.20	7.4	7.3	9.1	7.2	5.3	7.8
150 x 200	28.84	13.64	42.95	0.50	2.02	6.9	6.8	8.7	6.7	5.1	6.6
200 x 270	29.22	12.42	43.78	0.51	2.01	6.0	6.0	7.0	5.9	4.5	5.8
270 x 400	29.46	10.84	44.62	0.57	1.99	4.1	4.1	4.2	4.1	3.5	3.9
-400	29.56	5.96	46.07	1.24	2.61	20.5	20.7	11.4	21.3	37.9	25.6
Calculated Head	29.30	10.72	44.33	0.67	2.09	100.0	100.0	100.0	100.0	100.0	100.0

Table 3-3(c)

Reactor Feed 3 Sieve and Chemical Analyses

	Chemical Analysis					Component Distribution					
FRACTION (Mesh) Reactor Feed - 3	P_2O_5	Insol	CaO	MgO	I&A	% Wt.	P_2O_5	Insol	CaO	MgO	I&A
+14	29.53	11.08	43.77	0.42	2.39	0.1	0.1	0.1	0.1	0.1	0.1
20 x 28	28.67	13.04	42.69	0.43	2.15	0.4	0.4	0.5	0.4	0.3	0.4
28 x 35	28.77	12.98	42.94	0.43	2.11	4.4	4.4	4.9	4.4	3.3	4.5
35 x 48	28.77	13.43	42.77	0.43	1.91	8.4	8.3	9.6	8.3	6.2	7.6
48 x 65	28.24	14.97	41.86	0.42	1.89	11.6	11.4	14.9	11.4	8.4	10.5
65 x 100	27.90	15.88	41.36	0.42	1.85	14.5	14.0	19.8	14.0	10.5	12.8
100 x 150	28.09	15.55	41.61	0.42	1.92	8.4	8.1	11.2	8.1	6.0	7.7
150 x 200	28.58	14.34	42.44	0.42	1.94	8.5	8.4	10.5	8.5	6.2	7.9
200 x 270	29.21	12.47	43.19	0.42	1.94	8.1	8.1	8.6	8.1	5.8	7.4
270 x 400	29.64	10.98	44.02	0.44	2.14	5.7	5.9	5.4	5.9	4.4	5.9
-400	29.97	5.66	44.29	0.95	2.47	29.9	30.9	14.5	30.8	48.8	35.2
Calculated Head	28.95	11.66	42.90	0.58	2.10	100.0	100.0	100.0	100.0	100.0	100.0

Chemical analyses of the three pond water samples is given below in Table 3-4.

Table 3-4

Chemical Analysis of Pond Water Samples

			grams/liter				
Sample	pH	Redox	P_2O_5	CaO	MgO	F	SO_4
Pond Water 1	1.4	244	11.6	1.9	0.3	5.3	4.6
Pond Water 2	1.3	230	19.5	2.2	0.7	7.2	7.2
Pond Water 3	1.1	244	15.0	2.8	0.5	6.2	6.2

3.3 Mineralogical Analyses

Mineralogical testwork was undertaken in order to provide a basis for conceptualizing flowsheets by which flotation technology could be best inserted into existing phosphoric acid plants. To accomplish this objective, selective sieve fractions of each of the nine ground rock samples were delivered to the Florida Institute of Phosphate Research (FIPR) for mineralogical analyses. The selected fractions analyzed by FIPR are listed as follows:

Matrial Sample	Sample I.D.	Sieve (mesh) +48	48/100	100/200	200/400	-400
Concentrate	1, 2, and 3		X	X	X	X
Pebble	1, 2, and 3		X	X	X	X
Reactor Feed	1, 2, and 3	X	X	X	X	X

Mineralogical analyses performed by FIPR consisted of:

- X-ray diffraction (XRD) to identify mineral species
- Scanning electron microscopy (SEM) to determine liberation of mineral species.

The results of the mineralogical testwork are given in the report issued by FIPR on 12/20/94. A copy of the FIPR report is included as Appendix B.

3.4 Discussion of Results

The P_2O_5 distribution versus mass balance distribution in the -400 mesh size fractions for the nine ground rock samples (three reactor feed samples, three ground concentrate samples and three ground pebble samples) are shown in Figure 3.1. Analysis of the data shows that preferential grinding of the phosphate mineral component did not occur since the P_2O_5 and weight distributions in the -400 mesh size fraction are essentially the same.

Figure 3.2 shows the MgO distribution versus mass distribution in the -400 mesh size fractions for the nine ground rock samples. As anticipated, all rock samples exhibited some degree of preferential grinding of the dolomitic mineral component. An unusual result, however, was that the data appear to show two types of preferential grinding. Seven of the nine rock samples show MgO to mass distribution ratios of approximately 1.80 to 1.0. Two of the rock samples (reactor feed 1 and concentrate 2) show MgO to mass distribution ratios of about 1.13 to 1.0. Both of these rock samples contained the lowest analyzed head MgO content.

The CaO to P_2O_5 ratio in the heads compared to the CaO to P_2O_5 ratio in the -400 mesh size fraction for the nine ground rock samples are shown in Figure 3.3. The data show that there is no significant preferential grinding for rock samples with CaO to P_2O_5 head ratios of 1.50 to 1.0 or lower. As the CaO to P_2O_5 ratio in the heads increases, however, a greater percentage of CaO reports to the -400 mesh fraction.

The mineralogical testwork performed by FIPR on selected size fractions of the nine rock samples identified the major mineral components as francolite, quartz, and dolomite. Minor XRD traces of calcite were also identified. Essentially, no locked dolomite was found in the -400 mesh fraction. The degree of liberation of +400 mesh particles could not be readily determined.

Figure 3.1

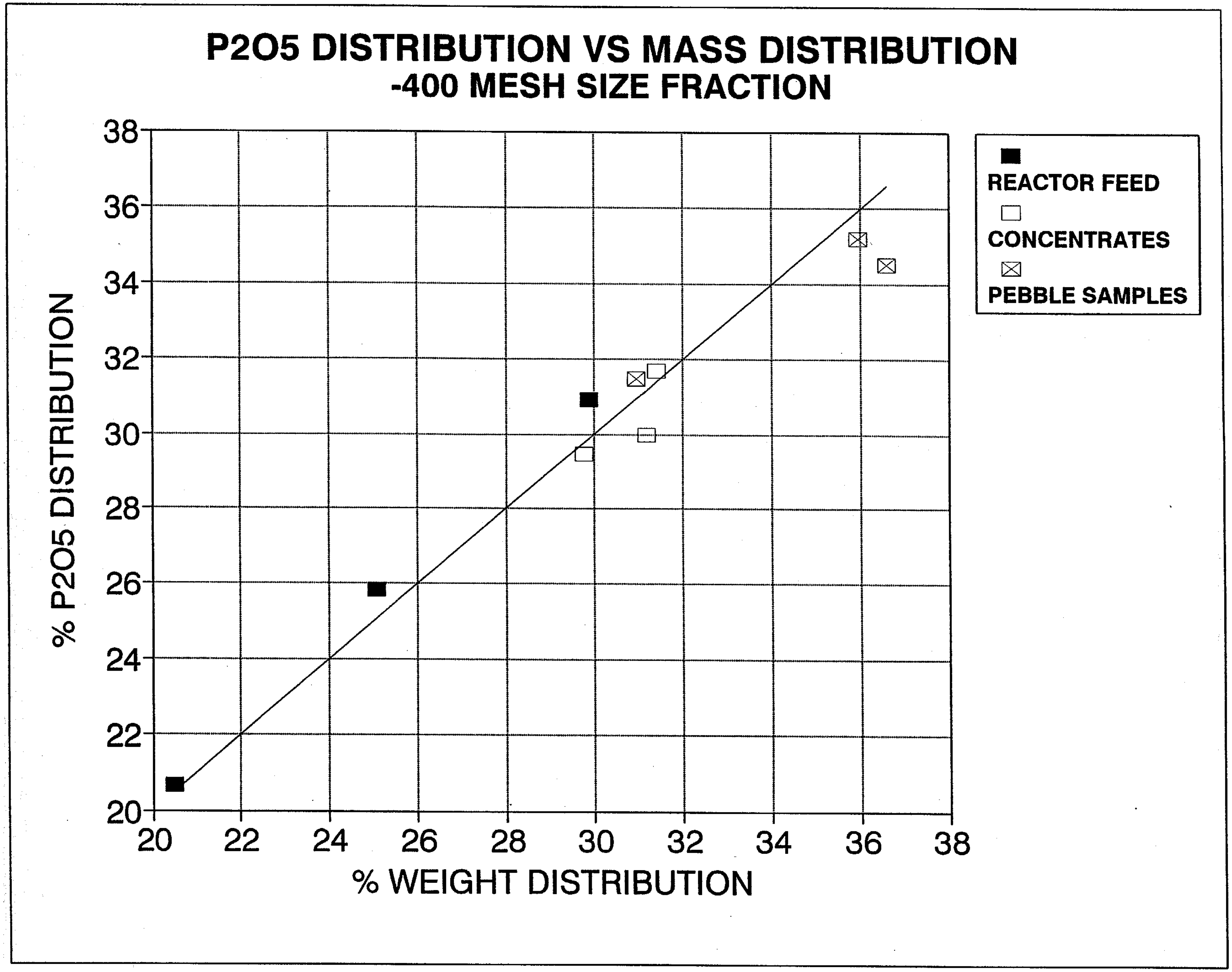

P2O5 DISTRIBUTION VS MASS DISTRIBUTION
-400 MESH SIZE FRACTION
% P2O5 DISTRIBUTION
% WEIGHT DISTRIBUTION
20
22
24
26
28
30
32
34
36
38
■ REACTOR FEED
□ CONCENTRATES
☒ PEBBLE SAMPLES

Figure 3.2

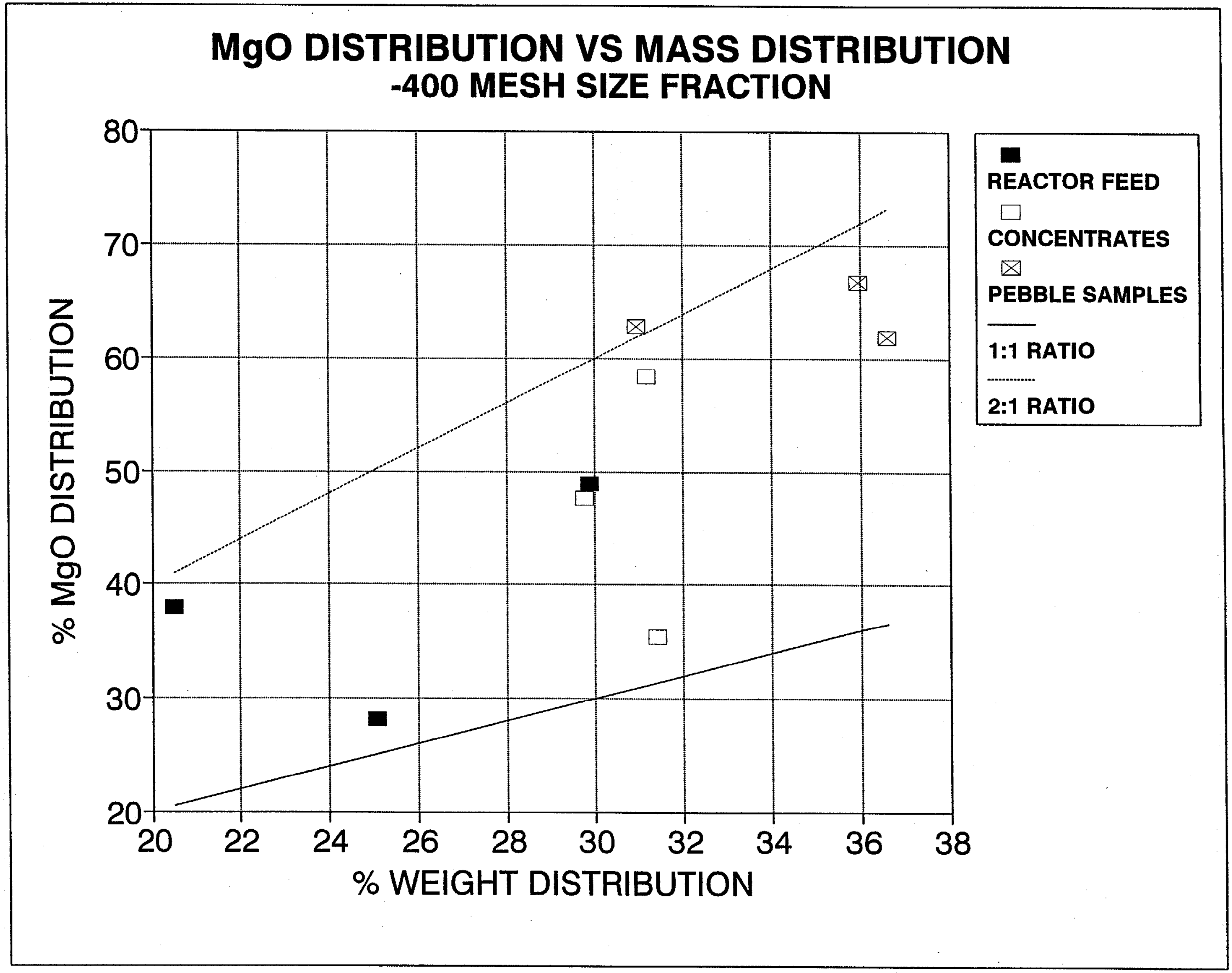
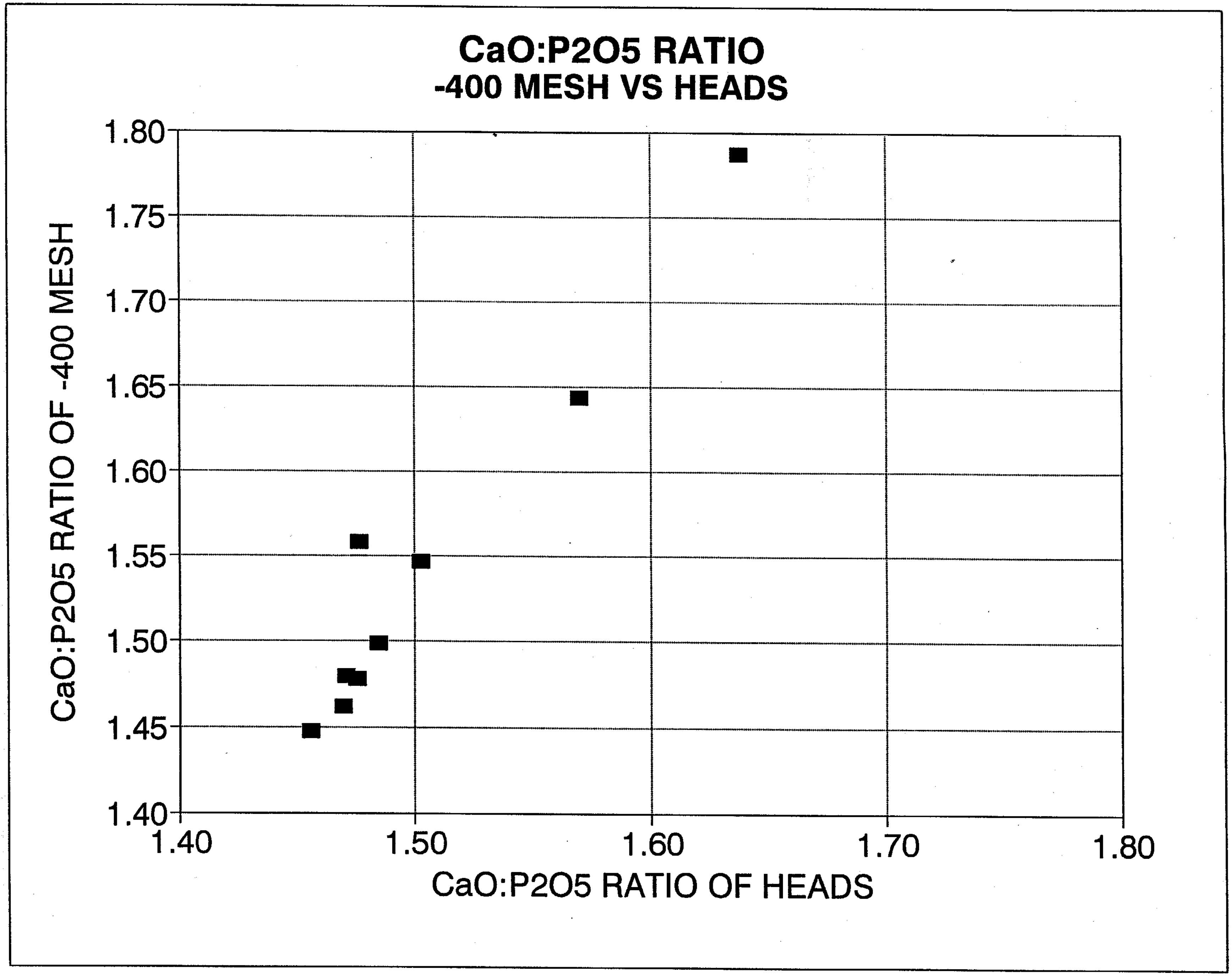

MgO DISTRIBUTION VS MASS DISTRIBUTION
-400 MESH SIZE FRACTION
% MgO DISTRIBUTION
80
70
60
50
40
30
20
% WEIGHT DISTRIBUTION
20
22
24
26
28
30
32
34
36
38
■ REACTOR FEED
□ CONCENTRATES
☒ PEBBLE SAMPLES
1:1 RATIO
2:1 RATIO

Figure 3.3


CaO:P2O5 RATIO
-400 MESH VS HEADS
CaO:P2O5 RATIO OF -400 MESH
1.80
1.75
1.70
1.65
1.60
1.55
1.50
1.45
1.40
1.40
1.50
1.60
1.70
1.80
CaO:P2O5 RATIO OF HEADS

SECTION 4
PROCESS EVALUATION TESTS

4.1 Objective

Flotation processes may be broadly categorized as direct or inverse, according to whether the concentrate or tailing is reagentized and removed as the froth product. For phosphate rock (reactor feed), the quantity of gangue is relatively small and conceptually inverse flotation schemes are preferred because it should be easier to reagentize and float the minor amount of tailings than to reagentize and float the major amount of concentrate.

The following three inverse flotation processes for removing carbonate gangue from ground phosphate rock were selected for evaluation:

- The BRGM process using a phosphoric ester collector.
- The BOM process using a tall oil collector.
- The IMC process using a sulfonated fatty acid collector.

The purpose of the evaluation task was to select, on the basis of comparative laboratory test results, a flotation process for removing calcite and dolomite gangue from ground phosphate rock.

4.2 BRGM Process

It is Jacobs understanding that the BRGM process was developed in cooperation with the Gerlund Company. French patents 79.30868 and 80.19366 were issued to BRGM, Henchiri et al, in 1979 and 1980. Additional information was published by Henchiri (1981, 1993).

In this process the phosphate rock is conditioned with a reagent suite comprising a phosphoric ester collector and a depressant (sulfuric acid and/or fluosilicic acid). During flotation, carbonate gangue minerals are removed as a froth product while quartz and phosphate are recovered as the cell underflow product. For Tunisian phosphate, the process has been more effective in removing carbonate particles in the 74 to 20 micron size range.

This process was tested on minus 400 mesh ground material from pebble samples 2 and 3, on plus 400 mesh ground pebble 3, and on the composite (plus and minus 400 mesh) ground pebble 3. Two different phosphoric esters (MDIT and SM-15) were tested as carbonate collector. Sulfuric acid and starch were used as phosphate depressants. Test results are summarized on Table 4-1, and laboratory report sheets listing test conditions and results are presented in Appendix C1.

The BRGM process was not successful in making a clean separation of carbonate minerals from ground phosphate rock produced in Florida. Concentrate of acceptable quality could not be recovered. It is possible that the particle size range of ground Florida phosphate rock exceeds the limitations of the BRGM process with regard to coarse (+74 microns) and fines (-20 microns). The majority of the liberated carbonates occur in the minus 400 mesh (-38 microns) fraction.

4.3 BOM Process

The Bureau of Mines Research Center at Albany, Oregon developed a flotation process for removing carbonate gangue from. western phosphate rock. Rule (1977, 1982) described laboratory and pilot scale tests and test results.

In this process phosphate rock ranging from 212 to 20 microns is conditioned with a tall oil collector in the presence of a fluosilicic acid depressant. During flotation the carbonate gangue minerals are removed with the froth while phosphate and silica are recovered as the cell underflow product. Bureau of Mines researchers reported that pond water could be successfully substituted for the fluosilicic acid depressant, and that the presence of minus 10 micron particles had a detrimental effect on flotation performance.

This process was tested on plus 400 mesh, minus 400 mesh, and composite material from ground pebble sample 3. Rather high quantities of collector are required to produce a froth product. Test results are summarized on Table 4-2, and laboratory report sheets listing test conditions and results are presented in Appendix C2.

Table 4-1

Summary Data - BRGM Process Evaluation Tests

Test	Feed	Collector(1)	H_2SO_4 (2)	% P_2O_5 Recovery	Concentrate Quality		
					% P_2O_5	% MgO	CaO:P_2O_5
6	-400 m, Pebble 2	MDIT - 3.0	(5.0) - 12.9	89.7	28.1	1.8	--
7	-400 m, Pebble 2	MDIT - 2.4	(5.0) - 13.9	85.9	28.1	1.8	--
8	-400 m, Pebble 2	MDIT - 4.7	(5.0) - 13.5	95.7	28.3	1.9	--
10	-400 m, Pebble 2	MDIT - 4.6	(5.0) - 15.2	87.8	28.2	1.8	
22	+400 m, Pebble 3	SMI5 - 1.2	(5.0) - 2.3	77.4	28.1	1.1	--
23	+400 m, Pebble 3	SMI5 - 0.3	(8.0) - 0	81.3	26.4	1.1	--
24	+400 m, Pebble 3	SMI5 - 0.3	(5.0) - 1.7	16.4	27.9	1.7	--
41	-400 m, Pebble 3	SMI5 - 0.5	(5.0) - 7.0	86.0	24.8	3.5	--
42	-400 m, Pebble 3	SMI5 - 0.6	(5.0) - 7.8	80.0	25.1	3.4	--
43	-400 m, Pebble 3	SMI5 - 0.7	(5.0) - 6.8	72.2	25.5	3.1	--
BRGM 12	Pebble 3	SMI5 - 0.2	(5.5) - 4.5	97.7	25.8	1.7	1.61
BRGM 13	Pebble 3	SMI5 - 0.3	(5.3) - 5.2	94.7	25.9	1.4	1.59
BRGM 14	Pebble 3	SMI5 - 0.4	(5.5) - 4.8	91.2	25.6	1.5	1.60
BRGM 15	Pebble 3	SMI5 - 0.5	(5.4) - 4.7	90.7	26.5	1.6	1.51
BRGM 16	Pebble 3	SMI5 - 0.35	(5.4) - 4.5	93.2	25.7	1.5	1.58
BRGM 17	Pebble 3	SMI5 - 0.5	- 1.9	94.0	26.1	1.4	1.55

(1) Phosphoric ester reagent - lbs/ton feed
(2) (pH) - lbs/ton feed

Note: Starting with test 10, starch was used in addition to H_2SO_4. In BRGM test 17, pond water was used instead of H_2SO_4 in the flotation cell.

Table 4-2

Summary Data - BOM Process Evaluation Tests

Test	Feed	Collector (1)	Depressant (2)	% P_2O_5 Recovery	Concentrate Quality		
					% P_2O_5	% MgO	$CaO:P_2O_5$
33	+400 m, Pebble 3	0.50	0.50	(3)	--	--	--
34	+400 m, Pebble 3	1.00	0.50	(3)	--	--	--
35	+400 m, Pebble 3	1.50	0.50	(3)	--	--	--
36	+400 m, Pebble 3	1.95	0.49	99.4	26.2	1.1	--
37	-400 m, Pebble 3	0.50	0.50	(3)	--	--	--
38	-400 m, Pebble 3	1.00	0.50	(3)	--	--	--
39	-400 m, Pebble 3	1.50	0.50	(3)	--	--	--
40	-400 m, Pebble 3	2.01	0.50	94.6	25.0	3.7	--
BOM 1	Pebble 3	0.99	0.49	98.9	26.0	1.9	1.60
BOM 2	Pebble 3	1.48	0.49	98.0	26.1	1.7	1.60
BOM 3	Pebble 3	1.97	0.49	97.6	25.4	2.0	1.62
BOM 4	Pebble 3	2.46	0.49	97.7	25.8	1.8	1.59
BOM 5	Pebble 3	1.43	0.48	99.6	26.9	1.6	1.50 (4)
BOM 6	Pebble 3	0.99	0.49	99.2	25.9	2.1	1.59
BOM 7	Pebble 3	1.48	0.49	98.4	25.6	1.7	1.61
BOM 8	Pebble 3	1.97	0.49	97.1	25.6	1.6	1.60
BOM 9	Pebble 3	2.47	0.49	96.8	26.0	1.6	1.59
BOM 10	Pebble 3	2.95	0.49	96.3	25.6	1.6	1.60
BOM 11	Pebble 3	2.92	0.49	99.5	26.9	1.5	1.53 (4)

(1) Emulsion (soap) - lb/ton feed
(2) Fluosilicic acid - lb/ton feed
(3) No flotation occurred
(4) Pond water used

The BOM Process was not successful in making a clean separation of carbonate minerals from ground phosphate rock produced in Florida. The P_2O_5 recoveries were high, but concentrate quality was not acceptable. The size limitations of the process are indicated as 65 mesh (212 microns) on the lower boundary. However, the process was unable to make a selective separation with or without the presence of fines.

4.4 IMC Process

Snow (1982) invented a flotation process based on the use of a sulfonated oleic acid collector for carbonate minerals and a sodium tripolyphosphate (STP) reagent for depressing the phosphate. In this process the phosphate rock slurry is adjusted to a pH of 5.5 to 6.0 and then conditioned with STP and the carbonate collector. During flotation the carbonate minerals are removed with the froth and phosphate plus any quartz are recovered as the cell underflow product.

This process was tested on minus 400 mesh ground phosphate rock from pebble samples 2 and 3, on plus 400 mesh from ground pebble 3, and on the composite ground pebble 3. The sulfonated oleic acid was tested with and without the presence of fatty acid collector The fatty acid can act as an extender and also as a defoaming agent.

The test results obtained from minus 400 mesh material from pebble 2 and pebble 3 samples are summarized on Table 4-3 and laboratory report sheets are presented in Appendix C3a.

The minus 400 mesh component of pebble 2, which included some clay material, was not responsive to flotation. The minus 400 mesh component of pebble 3, which was high in carbonate content, was also not responsive to flotation. Additional collector causes more material to be removed with the froth; however, the chemical analyses of the froth product and the cell product remain similar.

The test results obtained from plus 400 mesh material from ground pebble sample 3 are summarized on Table 44 and the laboratory report sheets are presented in Appendix C3b. The data indicate some response to flotation in that increasing the collector dosage causes more phosphate and carbonates to float: however, P_2O_5 recoveries remain high at 97 to 94%, while the concentrate carbonate mineral content is reduced

Table 4-3

Summary Data - Minus 400 Mesh Material
IMC Process Evaluation Tests

		Reagent Usage(1)			% P_2O_5	Concentrate Quality		
Test	Feed	Collector	H_2SO_4	STP	Recovery	% P_2O_5	% MgO	$CaO:P_2O_5$
1	Pebble 2	1.59 (2)	9.70	1.46	66.4	28.6	1.7	--
2	Pebble 2	3.16 (2)	9.46	1.45	41.3	29.4	1.2	--
3	Pebble 2	1.56 (2)	10.50	1.43	65.5	28.6	1.8	--
4	Pebble 2	1.46 (3)	9.79	1.43	51.9	28.7	1.5	--
5	Pebble 2	1.57 (2)	9.79	2.87	64.8	28.6	1.6	--
9	Pebble 2	1.62 (2)	9.91	1.49 (4)	63.2	28.6	1.7	--
18	Pebble 3	0.25 (2)	6.99	1.49	97.9	24.7	3.7	--
19	Pebble 3	0.50 (2)	6.97	1.49	95.3	24.8	3.7	--
20	Pebble 3	1.00 (2)	6.93	1.49	84.8	25.0	3.6	--
21	Pebble 3	1.97 (2)	7.19	1.50	71.2	25.4	3.3	--
29	Pebble 3	0.20 (3)	6.89	1.49	97.2	24.7	3.8	--
30	Pebble 3	0.41 (3)	8.08	1.50	88.8	24.8	3.7	--
31	Pebble 3	0.81 (3)	6.87	1.49	79.5	25.0	3.6	--
32	Pebble 3	1.62 (3)	7.19	1.50	71.5	25.6	3.2	--

(1) Lb/ton feed
(2) Collector is a 1:1 mix of active sulfonated oleic acid and fatty acid.
(3) Collector is active sulfonated oleic acid only.
(4) Starch (0.2 lb/ton feed) also added as a phosphate depressant.

Table 4-4

Summary Data - Plus 400 Mesh Material
IMC Process Evaluation Tests

		Reagent Usage(1)			% P_2O_5	Concentrate Quality		
Test	Feed	Collector	H_2SO_4	STP	Recovery	% P_2O_5	% MgO	$CaO:P_2O_5$
14	Pebble 3	0.24 (2)	2.20	1.45	99.1	26.0	1.1	--
15	Pebble 3	0.49 (2)	1.60	1.46	99.4	26.3	1.0	--
16	Pebble 3	0.97 (2)	1.62	1.45	98.6	26.2	0.9	--
17	Pebble 3	1.93 (2)	1.81	1.45	96.9	26.2	0.8	--
25	Pebble 3	0.20 (3)	3.07	1.46	99.3	26.2	0.9	--
26	Pebble 3	0.39 (3)	2.49	1.44	98.1	26.3	0.9	--
27	Pebble 3	0.79 (3)	1.86	1.45	95.8	26.4	0.8	--
28	Pebble 3	1.57 (3)	2.13	1.45	93.8	26.4	0.7	--

(1) Lb/ton feed
(2) Collector is a 1:1 mix of active sulfonated oleic acid and fatty acid.
(3) Collector is active sulfonated oleic acid only.

(0.7 to 0.8% MgO) only slightly. The data also indicate that adding fatty acid with the sulfonated oleic acid is not warranted.

Additional test results obtained from ground pebble sample 3 and from a ground blend of pebble sample 3 and concentrate sample 1 are presented on Table 4-5. Laboratory report sheets for these tests are given in Appendix C3c. For these tests, all material from the grinding mill was conditioned with reagents and floated.

For ground pebble 3, the IMC process made a more selective separation of phosphate from carbonate minerals than either the BRGM or BOM process. However, none of the three processes were able to upgrade pebble sample 3 to a commercially acceptable phosphate rock at the target P_2O_5 recovery of ≥ 95%.

The blend of pebble 3 and concentrate 1 had the following average head analyses:

	Avg.	Std. Dev.
% P_2O_5	28.03	± 0.17
% MgO	1.39	± 0.10
% CaO	43.33	± 0.25

This blend is not unlike the type of phosphate rock that may be produced from south Florida ore deposits. For the ground blend the IMC Process was able to reduce the carbonate content of the concentrate at relatively high % P_2O_5 recovery.

4.5 Discussion of Results

Based on results of the process evaluation tests, the IMC Process was superior to either the BRGM or BOM processes for removing carbonate minerals from ground phosphate rock produced in Florida.

Most of the liberated carbonate minerals are concentrated in the minus 400 mesh fraction of the ground phosphate rock; however, all three processes evaluated were ineffective at treating only the minus 400 mesh material.

The plus 400 mesh component of the ground phosphate rock does not contain sufficient liberated carbonate minerals to warrant being processed separately. As neither the plus

Table 4-5

Summary Data - Ground Phosphate Rock
IMC Process Evaluation Tests

		Reagent Usage(1)			% P_2O_5	Concentrate Quality		
Test	Feed	Collector	H_2SO_4	STP	Recovery	% P_2O_5	% MgO	$CaO:P_2O_5$
REJ-44	Pebble 3	0.39	4.40	1.45	97.2	26.1	1.2	--
REJ-45	Pebble 3	0.79	5.11	1.46	91.4	26.3	0.9	--
REJ-46	Pebble 3	1.60	4.87	1.48	84.7	26.2	0.9	--
REJ-47	Pebble 3	0.80 (3)	3.75	1.48	91.0	26.3	0.7	--
IMC 18	Blend (4)	0.40	5.66	1.47	97.2	28.9	0.9	1.51
IMC 19	Blend (4)	0.60	5.78	1.47	94.5	29.0	0.9	1.52
IMC 20	Blend (4)	0.80	6.18	1.47	92.1	28.7	0.8	1.52
IMC 21	Blend (4)	1.00	6.47	1.47	91.2	28.9	0.8	1.52
IMC 22	Blend (4)	0.94 (5)	2.44	1.39	92.2	29.1	0.8	--
IMC 23	Pebble 3	0.40	5.09	1.46	96.2	26.6	1.30	1.60
IMC 24	Pebble 3	0.60	4.68	1.47	93.8	28.4	1.10	1.50
IMC 25	Pebble 3	0.79	4.79	1.47	91.9	26.7	1.00	1.58
IMC 26	Pebble 3	0.99	4.86	1.47	90.9	26.9	1.00	1.57
IMC 27	Pebble 3	0.95 (5)	2.23	1.41	90.3	27.1	1.10	--

(1) Lb/ton feed
(2) Collector is a 1:1 mix of active sulfonated oleic acid and fatty acid.
(3) Collector is active sulfonated oleic acid only.
(4) Starch (0.2 lb/ton feed) also added as a phosphate depressant.
(5) Pond water used for flotation.

400 mesh or the minus 400 mesh can be effectively treated as separate fractions, it appears most expedient to process the composite mill discharge.

References (for Section 4)

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SECTION 5
FLOTATION PROCESS OPTIMIZATION

5.1 Objective

The purpose of the optimization tests was to examine two phosphate rock materials and determine which parameters of the IMC process could be changed to improve the separation of carbonate minerals from phosphate rock.

To achieve this objective, statistically designed tests were performed on ground pebble 3 and a ground blend of pebble 3 and concentrate 1. The composition of these two materials had the following analyses.

	Pebble 3		Blend	
	Avg.	Std. Dev.	Avg.	Std. Dev.
% P_2O_5	25.41	±0.40	27.66	±0.15
% CaO	41.50	±0.36	43.88	±0.26
% MgO	1.71	±0.07	1.29	±0.07

The effects of selected conditioning and flotation parameters were determined by statistically evaluating the following flotation responses:

- % P_2O_5 Recovery
- Concentrate $CaO:P_2O_5$ Ratio
- Coefficient of Separation for P_2O_5 and MgO = % P_2O_5 recovery - % MgO recovery

5.2 Ground Pebble 3

Twenty tests were performed on pebble 3. The first set of 16 tests examined pH level (5.0 vs 5.5), pH modifier (H_2SO_4 vs pond water), flotation pulp density (1.5 L cell vs 3.0 L cell), and conditioning time for the sulfonated oleic acid collector. The second set of four tests examined the pulp density during conditioning.

The results of the first set of 16 tests are summarized on Table 5-1 and the laboratory report sheets are presented in Appendix C4. For pebble sample 3 the pH level and pH

Table 5-1

Conditioning and Flotation Parameters
(four variables @ two levels factorial design)

	Effect of Changing from Level 1 to Level 2		
Variable	% P_2O_5 Recovery	$CaO:P_2O_5$ Ratio	Coef. Sep.
1) pH level	+0.4	+0.012	-0.4
2) pH modifier	+0.1 (2)	-0.007	-0.6 (2)
3) cell size (1)	***13.5***	-0.007	***+3.7***
4) conditioning time	-0.5	+0.006	***+1.4*** (2)
Interactions			
(1 x 2)	--	-0.002	+0.9
(1 x 3)	--	+0.004	-0.1
(1 x 4)	+0.1	***-0.016*** (2)	+0.3
(2 x 3)	+0.2	+0.007	***-1.5*** (2)
(2 x 4)	+0.3	+0.002	+0.2
(3 x 4)	+0.1	+0.007	+0.9
(1 x 2 x 3)	-0.1	+0.007	-0.5
(1 x 2 x 4)	-0.2	-0.006	--
(1 x 3 x 4)	-0.2	+0.003	-0.8
(2 x 3 x 4)	+0.1	-0.007	-0.1

(1) use of the 1.5 L cell corresponds to a liquid:solid ratio of 2:1, while the 3.0 L cell corresponds to a liquid:solid ratio of 5:1

(2) effect is statistically significant

	Level 1	Level 2
pH level	5.0	5.5
pH modifier	H_2SO_4	pond water
cell size (1)	1.5 L	3.0 L
conditioning time	30 sec.	90 sec.

modifier were not significant variables. The cell size (more dilute pulp) significantly increased the P_2O_5 recovery and improved the separation of dolomite from phosphate. Increasing the conditioning time from 30 to 90 seconds significantly improved the separation of dolomite from phosphate. The interaction between pulp pH and conditioning time had a small but significant effect on the concentrate $CaO:P_2O_5$ ratio. The most favorable ratio (1.567) was achieved at pH 5.0 and 30 seconds conditioning as shown in the following tabulation.

Table 5-2
$CaO:P_2O_5$ Ratios
(4 test averages)

	5.0 pH	5.5 pH
Conditioning time of 30 sec.	1.567	1.602
Conditioning time of 90 sec.	1.596	1.592

Similarly, the interaction between pH modifier and cell size (liquid:solid ratio) significantly influenced the coefficient of separation. The highest coefficients were obtained with H_2SO_4 and the 3.0 L cell (higher liquid:solid ratio) as shown on Table 5-3.

Table 5-3
Coefficient of Separation (P_2O_5 - MgO)
(4 test averages)

	H_2SO_4	Pond Water
2 liquid:1 solid	38.6	39.6
5 liquid:1 solid	43.9	41.8

The set of four tests examined conditioner pulp solids, from 35% to 65% at constant conditions, as follows:

sulfonated oleic acid	0.99 lb/ton feed
STP	0.73 lb/ton feed
pH level	5.0
pH modifier	pond water
conditioning time	90 seconds
cell size	3.0 L

Test results are presented on Table 5-4 and the laboratory reports are included in Appendix C4.

Table 5-4

Effect of Conditioning % Solids

Conditioning % Solids	% P_2O_5 Recovery	$CaO:P_2O_5$ Ratio	Coefficient of Separation	Concentrate % MgO
35	88.4	1.626	38.8	0.95
45	87.1	1.594	39.8	0.91
55	87.3	1.616	40.9	0.89
65	86.3	1.591	43.4	0.83

The above results clearly show that for pebble 3 the IMC process is improved by high solids conditioning, in particular the carbonate mineral rejection is enhanced with only a minor impact on P_2O_5 recovery.

5.3 Blend of Pebble 3 and Concentrate 1

Nine sets of tests were performed on the blend of pebble 3 and concentrate 1 to examine and re-examine conditioning and flotation parameters for the IMC process. A listing of the test sets follows.

Set	Tests	Variables Examined
1	16	STP dosage, conditioning % solids, pH modifier and flotation pH.
2	4	Conditioning % solids.
3	18	Conditioning % solids, conditioning time, and froth height.
4	6	Grinding with and without pond water.
5	4	Flotation % solids and froth height.
6	4	Grinding with and without pond water, with and without pH control in flotation.
7	6	Reagent addition schemes.
8	6	Flotation % solids and rotor rpm.
9	2	Flotation cell size.

Test Set 1 - A 2^4 factorial design was performed. The results are summarized on Table 5-5 and laboratory report sheets are given in Appendix C5. Average and optimum conditions for the testing are tabulated below.

	% P_2O_5 Recovery	$CaO:P_2O_5$ Ratio	Coefficient of Separation
16 test average	91.6	1.557	41.3
optimum	92.9	1.531	42.9

The test optimum exists at 1.5 lb STP/ton feed, 55% conditioning solids, using pond water instead of H_2SO_4, and controlling the flotation pH at 5.5. As shown on Table 5-5, the effects of the variables were small. However, the results show that it is possible to improve P_2O_5 recovery and concentrate quality simultaneously.

Test Set 2 - Four tests were performed to further examine the influence of conditioning percent solids. The slurry solids content in the conditioner were varied from 35% to 65%. Other conditions were held constant as follows.

sulfonated oleic acid	0.70 lb/ton feed
STP	0.75 lb/ton feed
pH level	5.0
pH modifier	pond water
conditioning time	90 seconds
cell size	3.0 L

Test results are presented on Table 5-6 and the laboratory reports are included in Appendix C5. The data confirmed the importance of high solids conditioning.

Table 5-5

Conditioning and Flotation Parameters (four variables @ two levels factorial design)

	Effect of Changing from Level 1 to Level 2		
Variable	% P_2O_5 Recovery	$CaO:P_2O_5$ Ratio	Coef. Sep.
1) STP dosage	***+0.8*** (1)	***-0.009*** (1)	--
2) Conditioning % solids	***-0.6*** (1)	***-0.012*** (1)	***+2.3*** (1)
3) pH modifier	***+0.7*** (1)	***-0.014*** (1)	***-1.0*** (1)
4) Flotation pH	***-1.1*** (1)	-0.001	+0.1
Interactions			
(1 x 2)	-0.1	+0.002	+0.5
(1 x 3)	***-0.3*** (1)	+0.002	-0.4
(1 x 4)	-0.1	--	-0.3
(2 x 3)	-0.1	***-0.005*** (1)	***+1.0*** (1)
(2 x 4)	***-0.3*** (1)	***+0.006*** (1)	-0.2
(3 x 4)	***+0.4*** (1)	-0.001	+0.2
(1 x 2 x 3)	***-0.3*** (1)	+0.002	+0.5
(1 x 2 x 4)	+0.1	-0.002	***-0.9***
(1 x 3 x 4)	+0.1	+0.001	+0.2
(2 x 3 x 4)	+0.1	***+0.006*** (1)	-0.4

Note: collector dosage was held constant at 0.8 lb/ton feed.

(1) Effect is statistically significant

	Level 1	Level 2
STP dosage	0.75	1.50
% solids	35	55
pH modifier	H_2SO_4	pond water
flotation pH	5.5	ambient (6.7)

Table 5-6
Effect of Conditioning Solids

Conditioning % Solids	% P_2O_5 Recovery	$CaO:P_2O_5$ Ratio	Coefficient of Separation
35	91.5	1.533	38.9
45	90.3	1.520	40.5
55	90.3	1.517	41.2
65	89.3	1.532	43.3

The test data show that the separation of carbonate minerals from phosphate is enhanced by high solids conditioning.

Test Set 3 - A central composite experimental design was planned and executed to examine first and second order effects of conditioning percent solids, conditioning time (seconds), and froth depth (inches) on flotation performance. The variable levels tested are coded below.

	-1.68	-1.00	0	+1.00	+1.68
% solids (X1)	45	49	55	61	65
Time (X2)	20	60	120	180	220
Froth depth (X3)	0.50	1.38	2.63	3.88	4.75

Conditions held constant during the tests were:

Sulfonated oleic acid	0.80 lb/ton feed
STP	0.75 lb/ton feed
pH level	5.0
pH modifier	pond water
cell size	5.0 L

Under the test conditions, % P_2O_5 recovery was not significantly influenced by any of the variables. The average P_2O_5 recovery was 96.2%.

The significant variable effects on the concentrate $CaO:P_2O_5$ ratio are demonstrated by the following equation, expressed as coded levels of the variables:

$$CaO:P_2O_5 = 1.534 - 0.003\,X2 + 0.003\,X3 - 0.008\,X1X3 - 0.006\,X2X3 + 0.004\,X2\,X2$$

Similarly, the next equation shows the effect of significant variables on the coefficient of separation (COS).

$$COS = 40.3 + 1{,}429\ X1 + 1.644\ X2 - 1.637\ X3 - 1.203\ X3\ X3$$

For the test conditions the maximum coefficient of separation is predicted when conditioning is conducted at 65% solids (1.68) for 220 seconds (1.68) and the froth depth during flotation is about 1.78 inches (-0.68). For these conditions, the coefficient of separation and $CaO:P_2O_5$ ratios are:

$$COS = 40.3 + 1.429(1.68) + 1.644(1.68) - 1.637(-.68) - 1.204\ (-.68)^2 = 45.9$$

$$CaO:P_2O_5 = 1.534 - 0.003(1.68) + 0.003(-0.68) - 0.008(1.68 \times -.68) - 0.006(1.68 \times -.68) + 0.004\ (-.68)^2 = 1.545$$

Relative to test set 1, the % P_2O_5 increased from 92.9 to 96.2, the $CaO:P_2O_5$ ratio increased slightly from 1.531 to 1.545, and the coefficient of separation increased from 42.9 to 45.9.

<u>Test Set 4</u> - Six tests were performed to examine if grinding with pond water changed the flotation response relative to grinding with tap water. Constant conditions for testing were as follows:

Sulfonated oleic acid	0.7 lb/ton feed
STP	0.75 lb/ton feed
pH level (1)	5.0
pH modifier	pond water
conditioning time	180 seconds
cell size	5.0 L

The test results are presented on Table 5-7.

Table 5-7

Effects of Grinding with Pond Water

	Water Source	% P_2O_5 Recovery	$CaO:P_2O_5$ Ratio	Coefficient of Separation
	tap	96.3	1.525	44.5
	tap	96.7	1.537	44.6
	tap	96.3	1.554	43.2
		96.4	1.539	44.1
	pond	96.3	1.540	46.2
	pond	95.4	1.531	46.2
	pond	95.5	1.521	46.7
		95.7	1.531	46.4
Difference in means		-0.7	-0.008	+2.3
Std. deviation of means		0.32	0.010	0.50

The data show an insignificant change in concentrate $CaO:P_2O_5$ ratio. The use of pond water in grinding appears to enhance the separation of carbonate minerals from phosphate and to slightly reduce the P_2O_5 recovery at constant collector dosage.

Test Set 5 - Four tests were performed to examine froth depth and flotation slurry percent solids (cell size). The 5-liter cell allows approximately twice the dilution as the 3-liter cell. For example, at 0.5 inch froth the corresponding percent solids are 9.5 and 18.3 for the 5-liter and 3-liter cells, respectively. At 2.5 inch froth the flotation pulp percent solids are 13.3 and 28.9 for the 5-liter and 3-liter cells, respectively. Test results are summarized on Tables 5-8, 5-9, and 5-10.

Table 5-8

P_2O_5 Recovery vs Cell Size and Froth Depth

	Cell Size		
Froth Depth (inches)	3-liter	5-liter	Avg.
0.5	94.6	96.8	95.7
2.5	90.6	96.6	93.6
Avg.	92.6	97.7	

The shallow froth (0.5 inch) and large cell resulted in highest P_2O_5 recoveries.

Table 5-9

$CaO:P_2O_5$ Ratio vs Cell Size and Froth Depth

	Cell Size		
Froth Depth (inches)	3-liter	5-liter	Avg.
0.5	1.531	1.511	1.521
2.5	1.485	1.509	1.497
Avg.	1.508	1.510	

The deep froth (2.5 inch) appeared to result in the lowest $CaO:P_2O_5$ ratio in the concentrate.

Table 5-10

Coefficient of Separation vs Cell Size and Froth Depth

	Cell Size		
Froth Depth (inches)	3-liter	5-liter	Avg.
0.5	42.8	38.9	40.8
2.5	42.7	36.6	39.7
Avg.	42.7	37.8	

The best separation was achieved with the smaller 3-liter cell. The coefficient of separation, similar to $CaO:P_2O_5$ ratio, did not appear to be influenced by froth depth. The least amount of floated material resulted when the large (5-liter) cell was used, possibly indicating inadequate agitation. Conditions held constant during the four tests were as follows.

sulfonated oleic acid	0.70 lb/ton feed
STP	0.75 lb/ton feed
pH level	5.0
pH modifier	pond water
conditioning	180 seconds @ 55% solids
cell rotor rpm	1100

Test Set 6 - Four additional tests were performed to confirm the effects of grinding with and without pond water and floating with and without pond water for pH control. Constant conditions for these four tests were as follows:

sulfonated oleic acid	0.80 lb/ton feed
STP	0.75 lb/ton feed
pH level	5.0
pH modifier	pond water
conditioning	120 seconds @ 65% solids

Test results are summarized on Table 5-11. The data confirm that grinding with pond water and pH control during flotation enhances the separation of carbonate minerals from phosphate.

Test Set 7 - A series of six tests were performed to examine variations in the scheme of reagent addition. In the standard procedure, grinding is performed at ambient pH and conditioning comprises three steps as follows.

- 60 second agitation with STP, followed by
- 15 second agitation with pH modifier, followed by
- 120 second agitation with collector.

Optimization testing indicated that 180 second conditioning with collector was beneficial and also that grinding with pond water (pH adjustment) prior to STP addition improved flotation response.

Four tests were performed to examine STP addition to the conditioner and two tests were performed to examine STP addition to the grinding mill instead of the conditioner. Test results are shown on Table 5-12. The data indicate that pre-conditioning the phosphate rock with STP prior to adding the collector serves no useful purpose. Adding STP and collector simultaneously with pH adjustment appears to be effective.

Table 5-11

Effect of Pond Water for Grinding and pH Control

	Pond Water for Grinding (1)		
% P_2O_5 Recovery	With (2)	Without	Average
slurry at ambient pH (3)	92.3	93.4	92.8
pH maintained at 5.0	93.1	93.3	93.2
Average	92.7	93.3	
$CaO:P_2O_5$ Ratio			
slurry at ambient pH (3)	1.509	1.521	1.515
pH maintained at 5.0	1.496	1.511	1.504
Average	1.503	1.516	
Separation Coefficient			
slurry at ambient pH (3)	49.0	46.7	47.8
pH maintained at 5.0	49.8	46.6	48.2
Average	49.4	46.6	

1) Grinding at 65% solids.

2) Pond water addition caused mill discharge to have a pH of about 5.

3) No pH modifier added to flotation cell.

Table 5-12
Effect of STP Addition

	Test Number					
	33 (1)	34 (1)	35 (2)	36 (2)	37 (2)	38 (2)
Conditioning Time (seconds)						
collector	180	180	180	180	180	180
STP	405	405 (3)	225	210	180	265 (4)
Flotation Response						
% P_2O_5 recovery	91.2	92.4	94.2	93.5	93.7	93.2
$CaO:P_2O_5$ ratio	1.508	1.498	1.502	1.483	1.504	1.483
Coefficient of Separation	48.1	46.4	46.5	48.7	48.6	48.5

(1) STP added to grinding mill (225 seconds)
(2) STP added to conditioner
(3) STP dosage is 0.376 lb/ton feed for test 34 only; for the other six tests, the dosage is 0.75 lb/ton feed.
(4) Normal procedure with three conditioning steps is 60 seconds STP followed by 15 seconds pH modifier, followed by collector conditioning.

Note: STP time is inclusive of agitation time for reagents added subsequently.

Test Set 8 - Six tests were performed to examine flotation percent solids and cell rotor rpm. The purpose of these tests was to supplement test set 5. The test results are summarized on Table 5-13.

Table 5-13
Effect of Cell Solids and Cell Rotor Speed

	Cell	Flotation Cell Slurry % Solids	
Performance Indicator	RPM	22 (3 L)	11 (5 L)
% P_2O_5 Recovery	1100	93.6	97.3
% P_2O_5 Recovery	1300	90.2	96.0
% P_2O_5 Recovery	1500	89.4	95.9
		91.1	96.4
$CaO:P_2O_5$ Ratio	1100	1.504	1.502
$CaO:P_2O_5$ Ratio	1300	1.493	1.499
$CaO:P_2O_5$ Ratio	1500	1.484	1.512
		1.494	1.504
Coefficient of Separation	1100	49.3	36.3
Coefficient of Separation	1300	48.1	46.6
Coefficient of Separation	1500	48.5	46.2
		48.6	43.0

The data show that 1100 rpm is adequate for the S-liter cell and inadequate for the 5-liter cell. Adequate agitation in the 5-liter cell is attained at or above 1300 rpm. The higher percent solids appears to give a higher rejection of both phosphate and carbonates to the froth; however, separation of carbonate minerals from phosphate minerals also appears greater.

Test Set 9 - Two tests were performed to compare flotation cell size at constant percent solids. Test results are shown on Table 5-14.

Table 5-14
Effect of Flotation Cell Size

Cell size	3 L	5 L
Rotor rpm	1100	1500
Slurry % solids	22	22
% P_2O_5 recovery	92.4	93.2
$CaO:P_2O_5$ ratio	1.50	1.50
Coefficient of separation	50.0	46.3

The data indicate that at comparable conditions the 5-liter cell does not separate carbonate minerals from phosphate rock as well as the 3-liter cell.

5.4 Discussion of Results

The optimization tests demonstrated improved performance for both pebble 3 and the blend of pebble 3 and concentrate 1; however, acceptable quality phosphate rock could not be extracted from pebble 3 at satisfactory P_2O_5 recovery. For the blended phosphate rock, improvements were made in terms of performance and reagent consumption, as shown on Table 5-15.

Table 5-15
Optimization Test Result Summary
(Blended Rock Sample)

	Feed Stock	Initial Results	Improved Results
% P_2O_5	27.66	28.44	28.95
% CaO	43.88	44.48	43.46
% MgO	1.29	0.74	0.62
% P_2O_5 Recovery	100	91	93
CaO:P_2O_5 Ratio	1.586	1.564	1.501
Lb reagent/ton feed			
STP	--	1.13	0.75
H_2SO_4	--	4.60	0 (1)
Collector	--	0.80	0.75

(1) H_2SO_4 has been replaced by pond water for pH control.

The performance improvement resulted from the following:

- increasing the percent solids in the conditioner (65% solids)
- increasing the collector conditioning time to 3 minutes
- substituting pond water for H_2SO_4 and using pond water for pH control during grinding, conditioning, and flotation (pH = 5.0 to 5.5).

Froth depth, flotation cell size, and rotor rpm also influence flotation performance.

The mesh-by-mesh recoveries for P_2O_5 and MgO are shown on Figure 5.1. The CaO:P_2O_5 ratios of tailings and concentrate mesh fractions are shown on Figure 5.2. These data show that dolomite rejection (MgO) is greatest in the minus 200 mesh fractions. Similarly, phosphate losses are greatest for the minus 400 mesh fraction.

Figure 5.1

RECOVERY FROM FEED TO CONCENTRATE

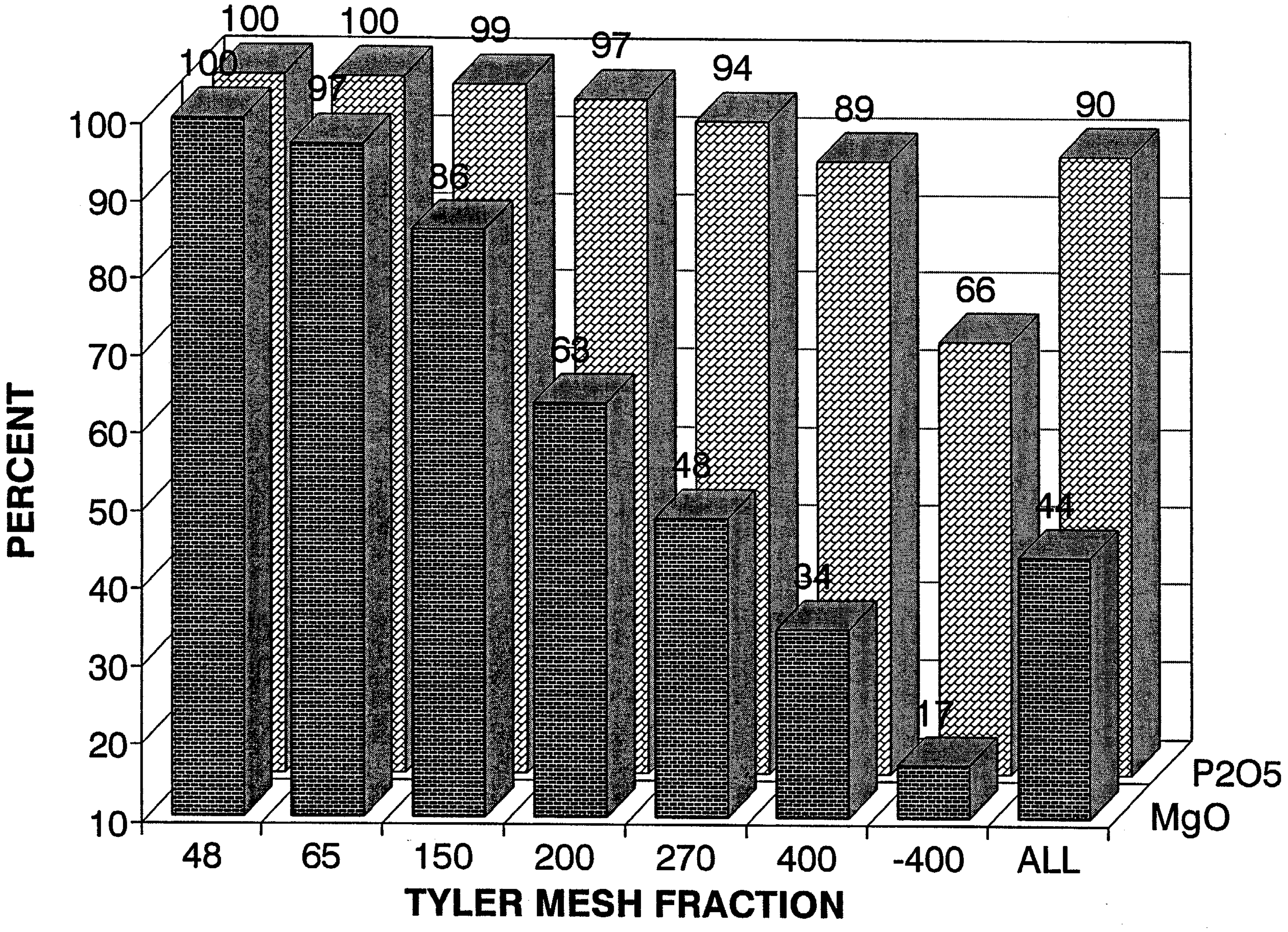
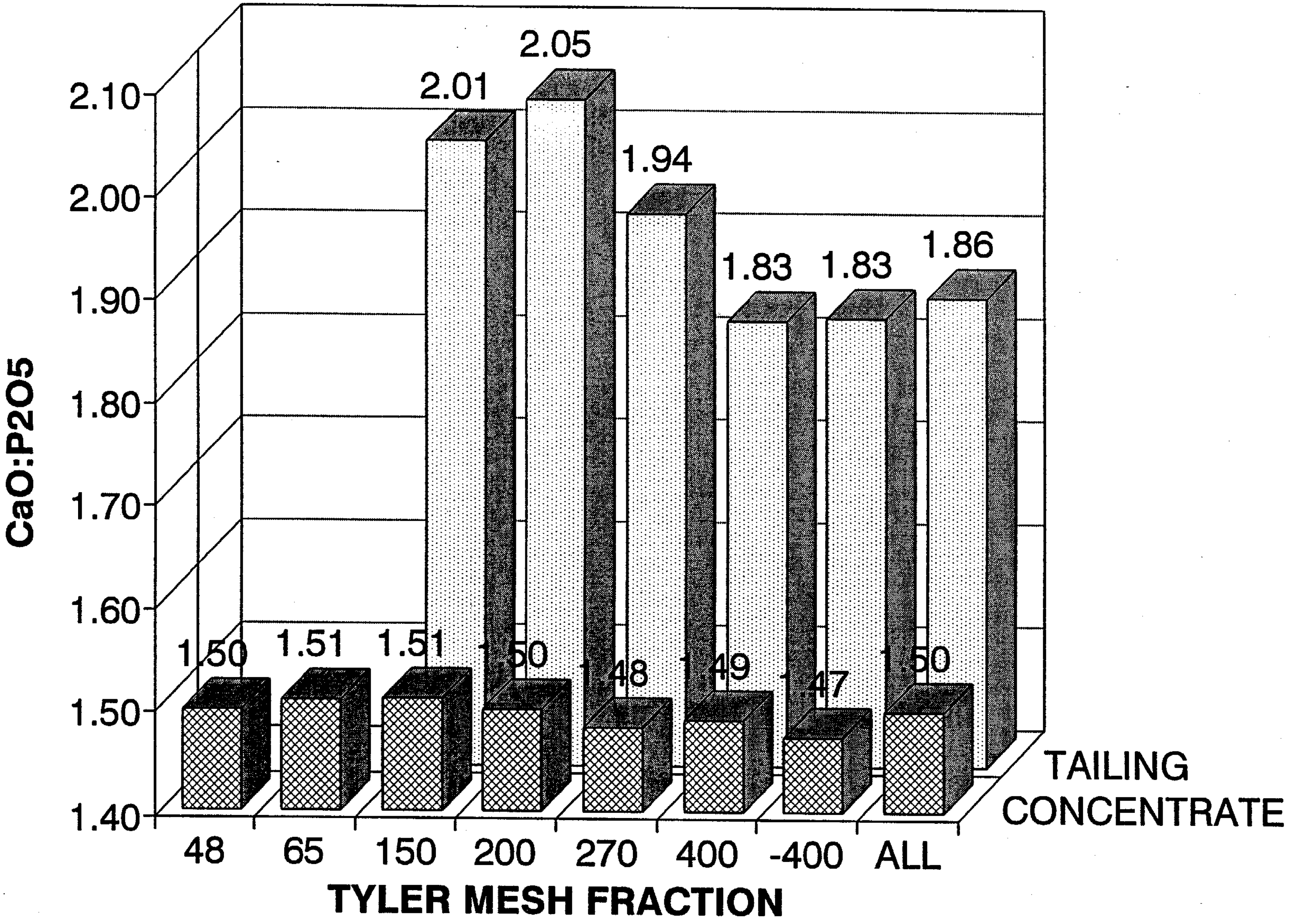


Figure 5.2

CaO:P2O5 RATIO



SECTION 6
FLOWSHEET CONFIRMATION

6.1 Objective

Flowsheet confirmation tests were conducted at the optimum levels of grinding, conditioning and flotation parameters identified from the process optimization tests results described in Section 5. The flowsheet confirmation tests consisted of two series of locked cycle tests to examine the effect of tailings and concentrate water recycle. The tests were performed on a ground blend of pebble 3 and concentrate 1.

The confirmation test results were used to develop a conceptual process flowsheet and materials balance for a flotation module added to a 1000 ton P_2O_5 per day phosphoric acid plant, as well as provide data for sizing equipment for the flotation module.

6.2 Locked Cycle Tests

The purpose of a locked cycle test is to simulate the effect of recycle streams in a continuous process by batch testing. The tests were specifically designed to evaluate the effects of recycling both tailings water and concentrate water.

A process block flow diagram depicting the locked cycle test configuration is given in Figure 6.1. Figure 6.1 shows the major process stages (grinding, reagent conditioning, anionic flotation, and dewatering of product and waste streams), quantities of solids, process water, recycle water and fresh water, and analytical requirements for the locked cycle tests.

Two locked cycle test series were performed. The first test series consisted of 30 cycles. For each cycle after cycle 11, 0.004 lb/ton of flocculant was added to the flotation concentrate prior to filtration. Filtrate water (containing residual flocculant) was then added to the carbonate tailings to facilitate dewatering. Decanted carbonate tailings water was then recycled to grinding and flotation. The second locked cycle test series consisted of 10 cycles and no flocculant was used.

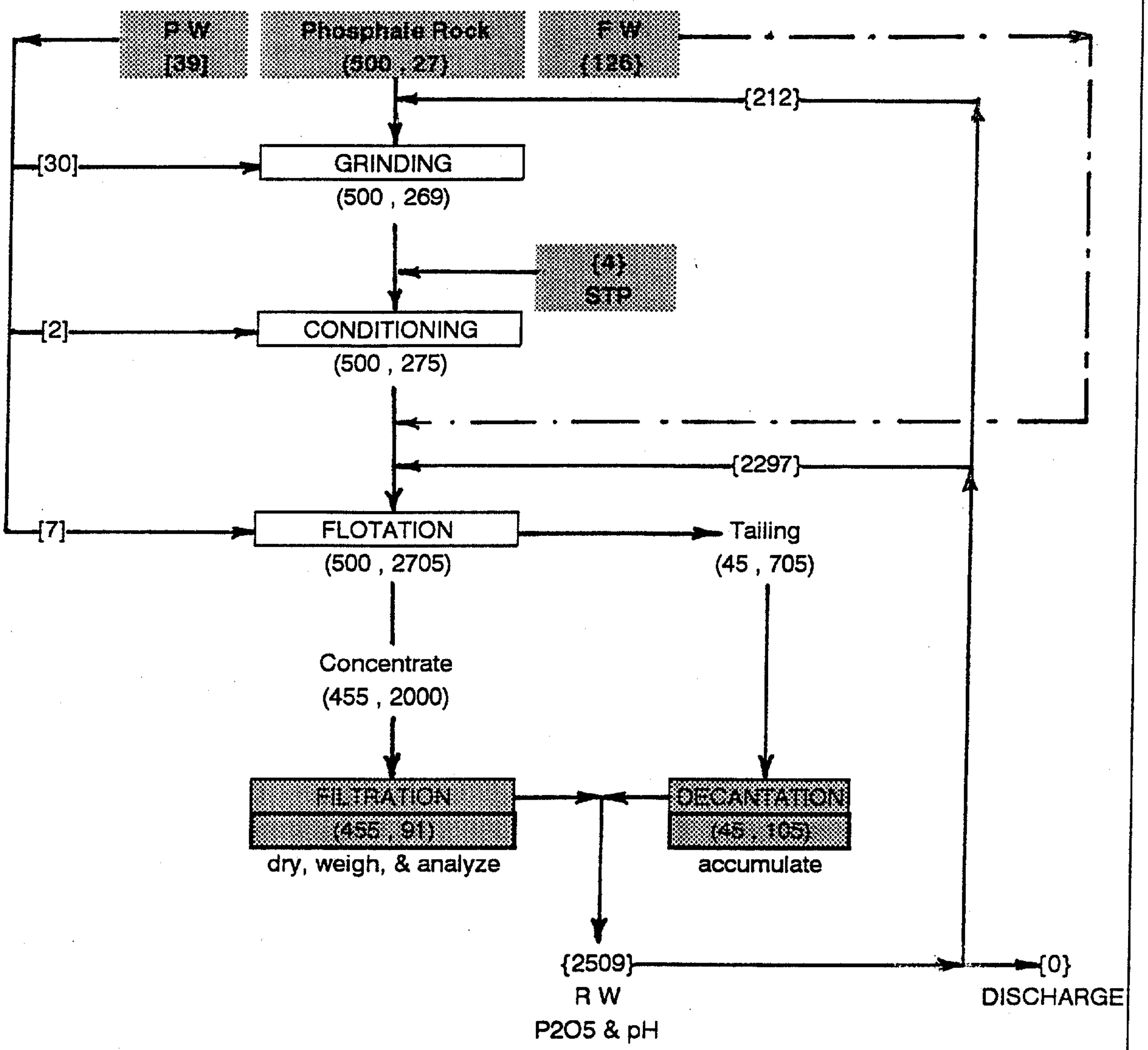


Figure 6.1

CYCLE TEST CONCEPT

LEGEND

{F W} = ML MAKEUP WATER
{R W} = ML RECYCLE WATER (pH ~ 6.5)
[P W] = ML POND WATER (pH 1.3)
(grams dry rock , ml liquid)

POND WATER ANALYSIS: P_2O_5,CaO,MgO,F,SO_4,& pH
FRESH WATER ANALYSIS: P_2O_5,CaO,MgO,F,SO_4,& pH
RECYCLE WATER ANALYSIS: P_2O_5,CaO,MgO,F,SO_4,& pH
P_2O_5 & pH each cycle
GROUND ROCK: P_2O_5,CaO,MgO,I&A,& Insol
CONCENTRATE: P_2O_5,CaO,MgO,I&A,& Insol
P_2O_5 & MgO each cycle
TAILINGS: P_2O_5,CaO,MgO,I&A,& Insol

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Results from all 30 cycles for the first test series are summarized in Table 6-1. Averaged data with an without flocculant are given in Table 6-2.

Table 6-2

Summary of Results
Locked Cycle Test Series 1

Cycles	(lb/ton)	P_2O_5	MgO	CaO	CaO:P_2O_5	P_2O_5	MgO	COS (1)
1-11	None	28.89	0.73	43.52	1.506	90.19	48.68	41.51
12-30	0.004	28.92	0.78	43.68	1.510	88.21	50.27	39.74
Difference (2)		+0.03	+0.05	+0.16		-1.98	+1.59	-1.77

(1) coefficient of separation
(2) with flocculant minus without flocculant

Although product quality remained essentially unchanged for the 30 test cycles the data show that flocculant use had a negative impact on separation efficiency. The average concentrate P_2O_5 recovery decreased from 90.19% to 88.21%, while the average MgO recovery increased from 48.68% to 50.27%, without and with flocculant, respectively.

As a result of the flocculant effect in the first locked cycle test series, a second locked cycle test series consisting of 10 test cycles was performed. Flocculant was not used in the second locked cycle test series.

In an effort to further improve the flotation concentrate grade and P_2O_5 recovery, some grinding, conditioning, and flotation parameters were adjusted for the second locked cycle test series. Table 6-3 summarizes the values of the test parameters for both locked cycle test series.

Table 6-1

Lock Cycle Flotation Test Results - Series 1

Test No.	Concentrate Analysis(%)			CaO:P_2O_5	Recovery		C.O.S.	Recycle Water
	P_2O_5	MgO	CaO		P_2O_5	MgO		P_2O_5(ppm)
1	29.12	0.77	44.05	1.51	92.40	51.88	40.52	200
2	28.74	0.76	43.88	1.53	87.05	48.88	38.17	183
3	28.88	0.73	43.38	1.50	92.01	49.39	42.62	151
4	28.83	0.64	42.88	1.49	89.03	41.97	47.06	246
5	29.12	0.70	43.88	1.51	90.05	45.97	44.08	294
6	28.85	0.70	43.05	1.49	90.12	46.43	43.69	302
7	29.08	0.70	43.55	1.50	90.67	46.35	44.33	281
8	28.94	0.74	43.21	1.49	91.31	49.58	41.73	331
9	28.43	0.73	42.88	1.51	88.69	48.36	40.33	299
10	28.94	0.77	44.22	1.53	90.59	51.18	39.41	293
11	28.85	0.79	43.72	1.52	90.10	52.39	37.71	290
12	28.69	0.68	43.05	1.50	86.69	43.63	43.06	130
13	28.83	0.72	43.38	1.50	89.34	47.38	41.96	147
14	28.78	0.72	43.21	1.50	89.96	47.79	42.17	201
15	28.88	0.72	43.55	1.51	88.50	46.85	41.65	215
16	29.02	0.78	43.88	1.51	91.44	52.19	39.25	221
17	29.12	0.77	43.96	1.51	90.55	50.84	39.71	224
18	28.93	0.80	43.63	1.51	89.38	52.48	36.90	239
19	28.74	0.81	43.46	1.51	87.36	52.28	35.08	268
20	29.30	0.81	44.05	1.50	88.43	51.91	36.52	189
21	28.97	0.77	43.88	1.51	88.20	49.78	38.42	205
22	29.16	0.85	43.96	1.51	88.05	54.50	33.55	197
23	29.16	0.80	43.88	1.50	88.68	51.66	37.02	210
24	29.02	0.78	43.88	1.51	88.25	50.37	37.88	206
25	28.74	0.80	43.72	1.52	88.38	52.24	36.14	214
26	29.35	0.79	44.38	1.51	88.27	50.45	37.82	155
27	28.42	0.82	43.05	1.51	86.00	52.69	33.31	165
28	29.03	0.78	43.88	1.51	87.83	50.11	37.72	179
29	28.66	0.77	43.22	1.51	85.64	48.86	36.78	186
30	28.70	0.78	43.88	1.53	85.09	49.10	35.98	171

Test No.	Water Analysis (ppm)				
	P_2O_5	MgO	CaO	F	SO_4
TAP	.69	15	67		49
Start Pond	15570	460	194	9600	7350
End Recycle	134	122	307	60	980

Test No.	Analysis (%)						
	P_2O_5	MgO	CaO	Fe_2O_3	Al_2O_3	F	Insol
Feed Head	28.03	1.32	44.22	1.15	1.03	3.28	10.09
Conc.#30	28.70	0.78	43.88	1.27	0.93	3.47	10.24
Tails#30	22.29	5.70	40.38	0.80	0.50	2.73	6.35
Tails#6-11	21.61	5.8					
Tails#12-29	22.43	5.5					6.07

NOTE: Tests 1-11, no flocculant used.
Tests 12-30, 0.004 lb/T flocculant used.

Table 6-3
Locked Cycle Test Parameters

Area	Parameter	Test Series 1	2
Grinding	% solids	65	65
	time (min.)	3.75	4.00
	pH	5.5	5.0
Conditioning	% solids	65	65
	prop.RPM	300	300
	STP dosage (lb/t)	0.75	0.75
	time (sec. w/STP)	15	15
	pH (for STP condit.)	5.5	5.0
	collector dosage (lb/t)	0.70	0.75
	time (sec. w/collector)	180	220
	pH (for collector condit.)	5.5	5.0
Flotation	cell size (liters)	3	3
	cell RPM	1500	1100
	froth depth (inches)	1.5	1.5
	flotation time (min.)	2	2
	pulp pH	5.5	5.0

Results from the second locked cycle test series are summarized in Table 6-4 and are illustrated in Figure 6.2. Essentially, equilibrium was attained after about seven cycles. Averaged performance data for the last three test cycles (cycles 9-11) compared to the baseline test are given in Table 6-5.

Table 6-5
Locked Cycle Test Summary

Test	Analysis (%)				CaO:	% Recovery	
Cycles	P_2O_5	MgO	CaO	P_2O_5	P_2O_5	MgO	COS (1)
Baseline	28.95	0.73	43.50	1.503	91.38	47.23	44.15
8-10	28.73	0.74	44.14	1.536	89.62	49.23	40.39

(1) coefficient of separation

Table 6-4

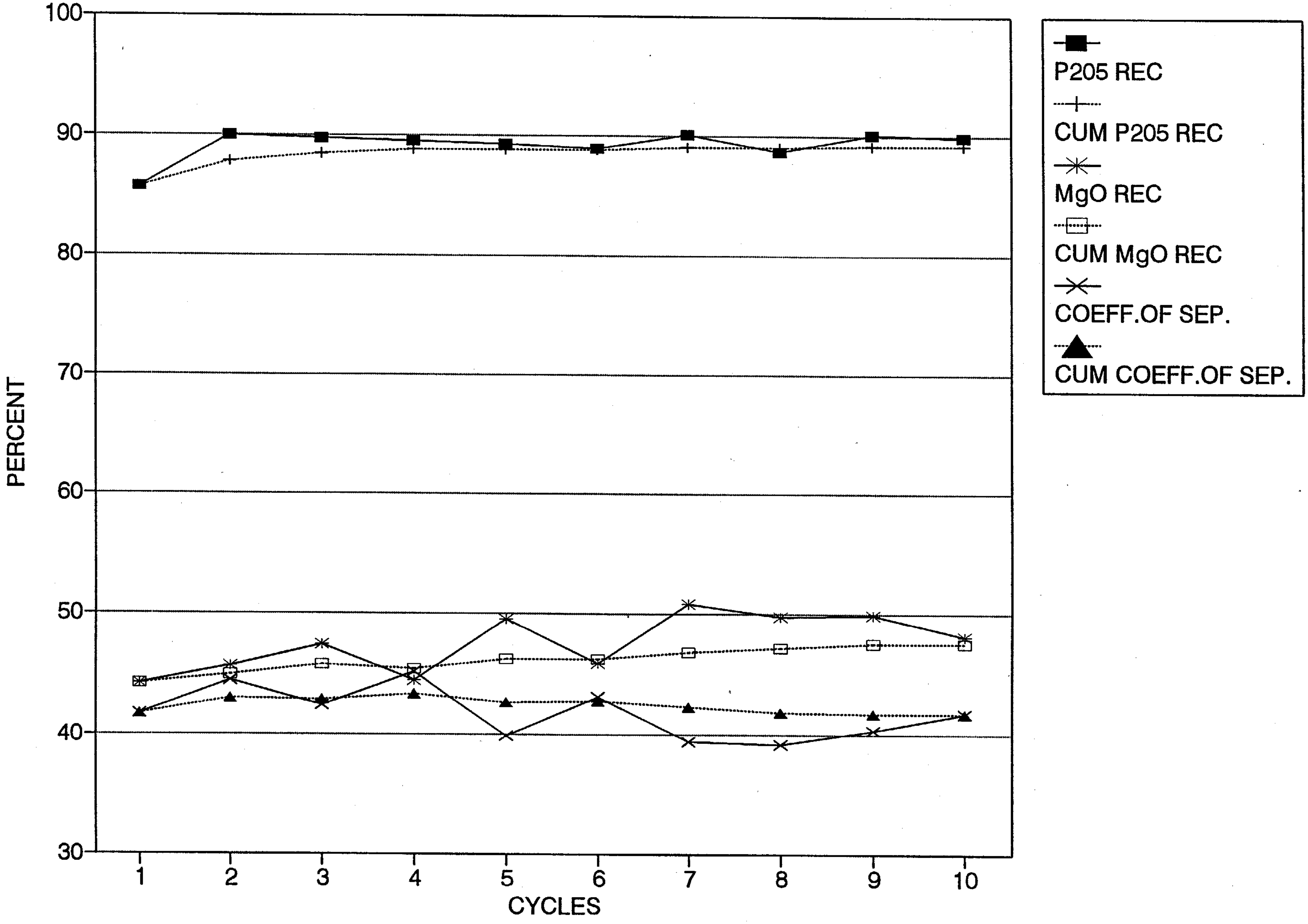
Lock Cycle Flotation Test Results - Series 2

	Concentrate Analyses (%)				% Recovery		
Test No.	P_2O_5	MgO	CaO	CaO:P_2O_5	P_2O_5	MgO	C.O.S.
1R	28.89	0.70	44.03	1.52	85.84	44.16	41.67
2R	28.94	0.69	43.95	1.52	90.07	45.60	44.47
3R	28.98	0.72	44.37	1.53	89.76	47.36	42.41
4R	29.08	0.68	44.54	1.53	89.57	44.48	45.10
5R	29.12	0.76	44.54	1.53	89.34	49.52	39.83
6R	28.84	0.70	44.20	1.53	88.94	45.84	43.10
7R	29.03	0.77	44.46	1.53	90.17	50.79	39.38
8R	28.84	0.76	44.37	1.54	88.79	49.69	39.11
9R	28.80	0.75	43.86	1.52	90.19	49.88	40.32
10R	28.56	0.72	44.20	1.55	89.89	48.12	41.77
Avg.	28.91	0.73	44.25	1.53	89.26	47.54	41.71

	Water Analysis (ppm)				
Test No.	P_2O_5	MgO	CaO	F	SO_4
TAP	.69	15	67	460	49
Start Pond	15570	460	194	9600	7350
End Recycle	328	200	240	80	1500

	Analysis (%)						
Test No.	P_2O_5	MgO	CaO	Fe_2O_3	Al_2O_3	F	Insol.
Feed Head	28.03	1.32	44.22	1.15	1.03	3.28	10.09
Tails#1-9	21.80	5.60	39.95	0.89	1.76		5.72
Tails#10	21.19	5.40	38.51	1.80	0.82	2.45	6.66
Conc.#10	28.56	0.72	44.20	1.39	0.87	3.49	10.64

FIGURE 6.2
LOCKED CYCLE TEST RESULTS - SERIES 2



The baseline test was performed using identical test parameter values and the locked cycle test, except that tap water was used instead of recycled concentrate and tailings water. The comparison indicates that recycle water use can have a slight adverse effect on process selectivity.

Settling tests were performed on the composite tails sample and concentrate from the first locked cycle test series. The tests were designed to provide data for sizing the concentrate thickener and estimating the water recovery from the carbonate tails settling area.

Data sheets for the flowsheet confirmation tests are given in Appendix C4.

6.3 Discussions of Results

6.3.1 General

The locked cycle tests demonstrated acceptable flotation performance compared to the baseline batch test. The results, however, showed that recycle water has a slight adverse effect on flotation selectivity. The use of flocculation to facilitate tailings dewatering also reduce flotation selectivity and is not recommended.

The concentrate sieve analysis for the two test cycles (tests 23 and 29) with the lowest coefficient of performance for the series 1 locked cycle test compared to the two process optimization batch tests (tests 45 and 46) with the highest coefficient of performance are presented in Table 6-6.

Table 6-6

Flotation concentrate Sieve Analysis Comparison

Test No.	Test Program	% +100 .esj	Coefficient Separation
23	locked cycle	40.8	37.02
29	locked cycle	41.2	38.78
Average		41.0	37.90
45	optimization	34.2	50.40
46	optimization	32.1	50.01
Average		33.1	50.20

The summarized data in Table 6-6 show that the bench test grinding was not very consistent. It also indicates that the flotation process is sensitive to grinding.

6.3.2 Mineral & Chemical Balance

A minerals and chemical component balance developed from the locked cycle test results and the characterization test data given in Section 3 are shown in Table 6-7. This balance was used as the basis for developing the flowsheet materials balance in Section 7. The flowsheet represents a carbonate flotation module installed in a 1000 tpd P_2O_5 phosphoric acid plant.

Table 6-7
Mineral and Chemical Component Balance

Group Component	Mineral Components Francolite	 Quartz	 Dolomite	 Calcite	 Total
phosphate	82.0	0	1.4	0	83.4
insol	0	10.0	0	0	10.0
dolomite	0	0	4.6	0	4.6
calcite	0	0	0	2.0	2.0
Total	82.0	10.0	6.0	2.0	100.0
mineral recovery	95.0(1)	100.0	52.0	48.0	91.98 (2)

Group Component	Chemical Components P_2O_5	 SiO_2	 MgO	 CaO
phosphate	28.0	0	0.30	41.17
insol	0	10.0	0	0
dolomite	0	0	1.00	1.40
calcite	0	0	0	1.12
Total Feed	28.0	10.0	1.30	43.69
concentrate assay	28.9	10.9	0.74	43.70

(1) The above balance is based on a coefficient of separation of 39%, which is slightly lower than the separation attained in the two sets of locked cycle tests. Although phosphate and dolomite recovery values differ from the locked cycle tests, the above separation is considered achievable.

(2) Total mineral recovery = (82.0)(.95)+(10.0)(1.00)+(6.0)(.52)+(2.0)(.48) = 91.98

6.3.3 P_2O_5 Balance

A 1000 tpd P_2O_5 phosphoric acid plant requires about 1075 tpd P_2O_5 in the phosphate rock fed to the reactor. The P_2O_5 balance, considering rock, pond water, and STP solution, for the envisioned conceptual process is given on Table 6-8.

Table 6-8

Conceptual Flotation Process - P_2O_5 Balance
(1000 tpd phosphoric acid plant)

	tons P_2O_5/24 hours		
	from rock	from solution	total
Phosphate rock (1)	1,131.9	0	1,131.9
Pond Water	0	4.9	4.9
STP solution	0	0.9	0.9
P_2O_5 inputs to flotation	**1,131.9**	**5.8**	**1,137.7**
Flotation tails (2)	56.6	4.1	60.7
Reactor feed (3)	1,075.3	1.7	1,077.0
P_2O_5 outputs	**1,131.9**	**5.8**	**1,137.7**

(1) 88% solids & 12% moisture
(2) consolidated to 40% solids
(3) thickened to 68% solids

The P_2O_5 recoveries from the rock and from the solutions are 95% and 29%, respectively. The P_2O_5 recovered from solution exceeds the P_2O_5 added as STP (The STP dosage is 0.75 lbs/ton of phosphate rock, equivalent to 0.43 lbs P_2O_5).

6.3.4 Water Balance

The water balance projected for the envisioned flotation process system is summarized on Table 6-9 and detailed on Figure 7.1.

Table 6-9

Conceptual Flotation Process - Water Balance
(1000 tpd phosphoric acid plant)

	Water (USGPM)
Makeup water	223.4
Phosphate rock @ 12% H_2O	92.0
Pond water	52.6
STP solution	5.1
H_2O inputs to flotation	**373.1**
Flotation tails @ 60% H_2O	81.1
Reactor feed @ 32% H_2O	291.9
H_2O outputs from flotation	**373.0**

(1) Pond water use comprises 40.5 gpm to grinding for pH adjustment and 12.1 gpm to conditioning and flotation. The additional 12.1 gpm reflects about 30% greater usage than required only for grinding (100 x 12.1/40.5 = 29.9).

The 0.1 gpm difference between inputs and outputs results from rounding. The above balance assumes rainfall and evaporation are equal on an annualized basis.

SECTION 7

CAPITAL AND OPERATING COSTS

7.1 Objective

Estimates of the capital cost and operating cost of a flotation module added to a 1,000 ton P_2O_5 per day phosphoric acid plant are provided in this section.

7.2 Summary

The estimated capital required to construct the flotation module is $5.15 million. The order-of-magnitude grade estimate (±25 percent accuracy) includes the materials and equipment, and the cost of engineering, procurement and construction (EPC) for a flotation module to be constructed at a 1,000 ton P_2O_5 per day phosphoric acid plant in central Florida.

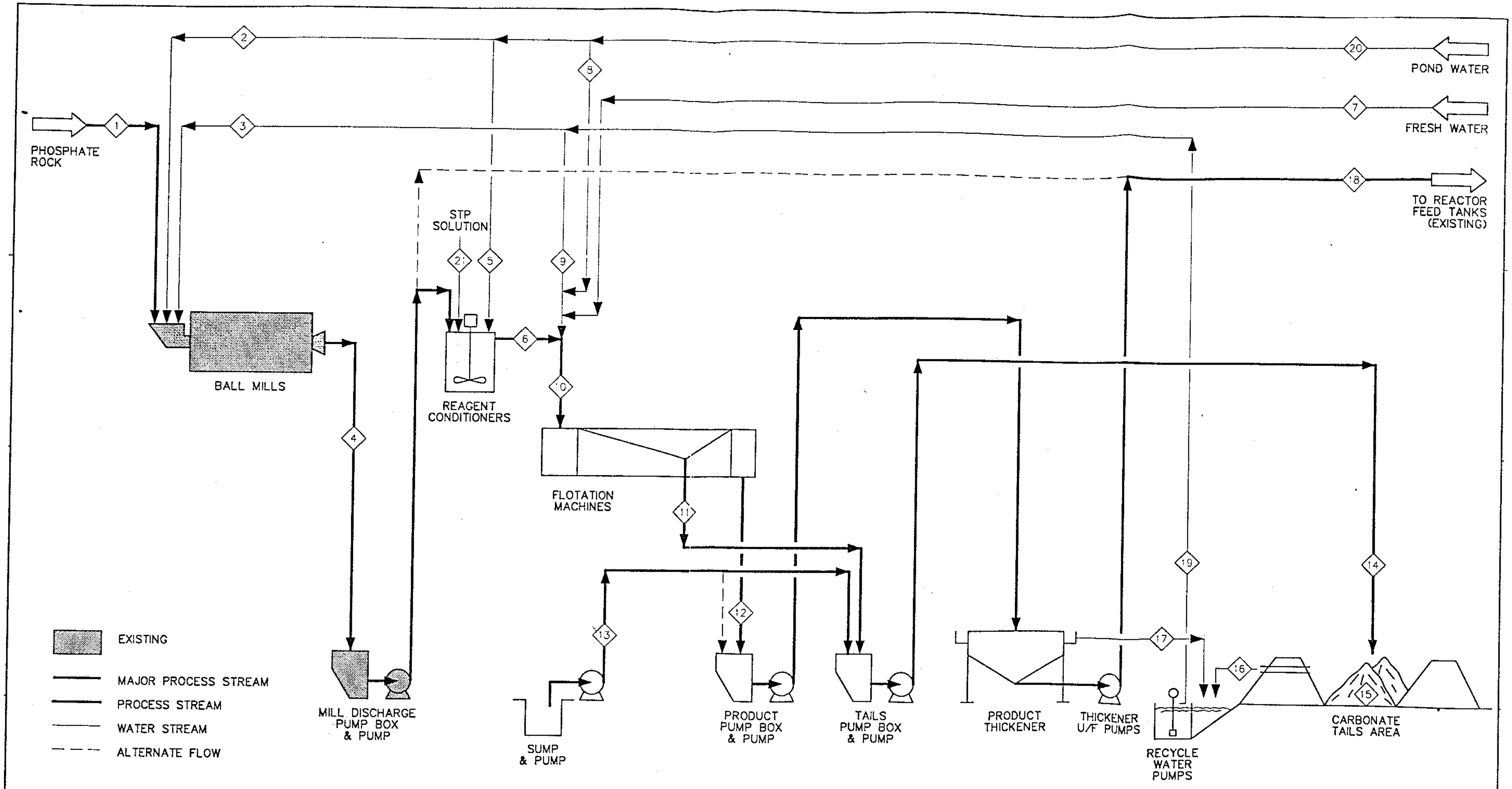
The estimated direct operating cost of the flotation module is $1.7 million per year, equivalent to $1.38 per ton of product, or $4.78 per ton P_2O_5.

7.3 Capital Cost Estimate

7.3.1 Basis

The order-of-magnitude estimate of capital required to construct the conceptual flotation module was based on in-house Jacobs cost factors and a priced equipment list developed from the process flowsheet and materials balance shown in Figure 7.1. The flotation module in Figure 7.1 is based on a compilation of results of the testwork described in the previous sections and consists of reagent conditioners, flotation cells, pump boxes and pumps, a product thickener, a reagent tank farm area, and a tailings disposal area: The complete equipment list for the flotation module is given in Appendix D.

The estimate is based on present-day pricing with no forward escalation included.



DESCRIPTION	STREAM NUMBER																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
TPH	168.4	0	0	168.4	0	168.4	0	0	0	168.4	13.5	154.9	–	13.5	13.5	0	0	154.9	0	0	0
% SOLIDS	88.0	0	0	67.4	0	66.9	0	0	0	15.9	4.7	20.0	–	4.7	40.0	0	0	68.0	0	0	0
GPM (WATER)	92.0	40.5	194.0	326.4	2.7	334.1	223.4	9.4	3009.2	3576.2	1095.3	2480.9	–	1095.3	81.1	1014.2	2189.0	291.9	3203.2	52.6	5.1
GPM (PULP)	N/A	40.5	194.0	580.8	2.7	588.6	223.4	9.4	3009.2	3830.6	1115.7	2715.0	–	1115.7	101.5	1014.2	2189.0	525.9	3203.2	52.6	5.1
SP. GR. (PULP)	N/A	1.032	1.000	1.723	1.032	1.714	1.000	1.032	1.000	1.110	1.030	1.142	–	1.030	1.332	1.000	1.000	1.734	1.000	1.032	1.017
TPH-P205 ROCK	47.161	–	–	47.161	–	47.161	–	–	–	47.161	2.358	44.803	–	2.358	2.358	–	–	44.803	–	–	–
TPH-P205 SOLUTION	–	0.157	0.006	0.164	0.010	0.212	–	0.037	0.101	0.349	0.107	0.242	–	0.107	0.073	0.034	0.073	0.169	0.107	0.205	0.037
TPH-P205 TOTAL	47.161	0.157	0.006	47.325	0.010	47.372	–	0.037	0.101	47.510	2.465	45.045	–	2.465	2.431	0.034	0.073	44.972	0.107	0.205	0.037
MgO TPH	2.205	0.005	0.006	2.215	0	2.216	–	0.001	0.092	2.309	1.095	1.213	–	1.095	1.065	0.031	0.067	1.146	0.098	0.006	0
CaO TPH	73.586	0.002	0.015	73.603	0	73.603	–	0	0.231	73.834	5.997	67.837	–	5.997	5.920	0.078	0.168	67.669	0.246	0.003	0

FIGURE 7.1

JE JACOBS ENGINEERING GROUP INC.
LAKELAND FLORIDA

PHOSPHATE ROCK TREATMENT FOR WASTE REDUCTION
FIPR CONTRACT No. 93-01-112R
CONCEPTUAL FLOTATION PROCESS PFD

PREPARED BY MEK
DRAWN BY CMP
DATE: 2-16-95
CADD NO. 621K001A
NONE
28-K621-00
KD-001

SAVE NAME: 621K001A
1 CMP
10/17/95 16:40

7.3.2 Estimated Constructed Cost

The estimated capital required to construct the flotation module is $5.15 million. The constructed cost is comprised of direct costs, indirect costs, professional services costs and includes a ten percent allowance for unforeseen costs and a four percent contractors fee. The components of the estimate are summarized on Table 7-1.

Direct costs include major process equipment, labor, subcontractors and equipment required for erection and installation. The labor component includes craft labor and supervision, but excludes fringes. Direct costs also include bulk commodity materials (concrete, piping structural steel, etc.), subcontracts (electrical supplies, paint, insulation, etc.) and construction labor for erection and installation.

Indirect costs include construction service labor, temporary facilities, craft fringes, payroll taxes and insurance, construction equipment, field staff and expenses.

Other costs include construction of the carbonate tailings settling area, professional services, expenses and contractors fee for engineering, procurement and construction. An allowance of ten percent for unforeseen costs has been added to the estimate due to the conceptual nature of the scope definition.

7.3.3 Exclusions

Items excluded from the estimated capital costs are:

- value for land
- permitting and other development costs
- inflation
- interest during construction
- startup costs
- working capital
- escalation
- all risk insurance
- liner for carbonate tailings settling area.

Table 7-1

Estimated Capital Requirements

	Total Cost ($)
DIRECT COSTS:	
Equipment	$954,000
Bulk Items	550,000
Labor	256,000
Sub-contracts	580,000
Sub-total Direct Costs	$2,339,000
INDIRECT COSTS:	
Field Indirects	320,000
Home Office Costs	630,000
Sub-total Indirect Costs	$950,000
OTHER COSTS:	
Tailings Dam	1,120,000
Sales/use tax (6%)	96,000
Allowance for Unforeseen (10%)	450,000
Contractors fee (4%)	198,000
Sub-total Other Costs	$1,864,000
TOTAL INSTALLED COST	$5,153,000

7.4 Operating Cost Estimate

7.4.1 Basis

The operating costs presented herein are categorized as direct (variable) costs and include the cost of delivered rock to the phosphoric acid facility, as well as the following cost elements for the flotation module:

- labor
- operating and maintenance supplies
- reagents
- electrical power.

The cost of pond water and fresh water were not included in the operating cost estimate. Indirect or fixed costs such as property tax, insurance, general overhead and administrative costs, depreciation, interest and capital payback are also not included in the operating cost estimate.

7.4.2 Direct Operating Costs

A summary of the estimated operating costs is given in Table 7-2. The total direct operating costs for the flotation module are $1.7 million per year, equivalent to $1.35 per ton of product, or $4.78 per ton P_2O_5. The total cost of rock to acidulation is $82.85 per ton P_2O_5 ($78.07 per ton P_2O_5 for delivered rock plus $4.78 per ton P_2O_5 for the flotation stage).

Labor costs are calculated for a work force of two people (operator and helper) operating three shifts per day of 8 hours each, with a fourth (swing) shift to accommodate weekends, holidays and vacations. Maintenance labor is estimated at one person at 2,080 hr/yr.

Operating and maintenance supplies are estimated by multiplying the process equipment capital cost by a percentage. These costs are for supplies required to maintain the process equipment and facilities in good condition. The factor used for the flotation module is 12 percent of the process equipment cost.

Table 7-2

Direct Operating Cost Summary

	Operating Cost		
Cost Element	Annual	Per ton Product	Per P_2O_5 Ton
· Delivered rock to phosacid(1)	$29,183,000	$23.79 (2)	$78.07
· Flotation Module			
·· Labor	292,000	0.24	0.82
·· Operating & Maintenance Supplies	114,000	0.09	0.32
·· Reagents	1,097,000	0.89	3.08
·· Electrical	200,000	0.16	0.56
Sub-total Flotation	$1,703,000	$1.38	$4.78
· **Total Operating Cost to Phosphoric Acid**	$30,884,000	$25.17	$82.85

(1) Annual rock received = (168.4)(24)(330) = 1,333,700 tons (373,800 P_2O_5 T)
Annual concentrate produced = (154.9)(24)(330) = 1,226,800 tons (356,400 P_2O_5 T)

(2) TFI rock cost = $21.88/ton rock received = ($21.88)(1,333,700/1,226,800) = $23.79/ton product

Reagent costs are calculated from the dosage per ton of flotation concentrate produced (based on the locked cycle tests described in Section 6.3). Calculations were made by multiplying the dosage per ton x reagent unit cost x tons per annum.

Electrical power costs are calculated from the rated motor horsepower as shown on the equipment list. The determination of horsepower hours per annum is made by multiplying the installed horsepower x the operating hours per year x a 0.88 load factor adjustment. The load factor adjustment is calculated as follows:

$$\text{load factor adjustment} = \frac{(\text{load factor})(\text{lighting factor})}{(\text{power factor})}$$

$$= \frac{(0.75)(1.05)}{(0.90)} = 0.88$$

The power cost of $0.043 per kWh was obtained from the TFI production cost survey for 1993 (Schedule 34 - weighted average of all reporting phosphoric acid plants).

SECTION 8
RECOMMENDATIONS

8.1 Potential of the Process

The flotation process for rejection of carbonates that was demonstrated from the test program yields three potential advantages to the industry: (1) higher consumption of pond water; (2) reduction in gypsum production; and (3) lower overall cost of DAP produced.

The process material balance on Figure 7.1 shows about 81 gpm water consumption with the flotation tailings, This additional water consumption due to the flotation process amount to 0.45 tons water per ton of P_2O_5 in the reactor feed.

The use of pondwater in grinding and as a pH modifier is beneficial to the flotation process. As pond water is conventionally used in grinding mill, the additional consumption of pond water is that amount used for pH control in conditioning and flotation. The increase in pond water consumption due to flotation is 0.07 tons of pond water per ton of P_2O_5 in the reactor feed. Relative to the quantity used in grinding, this is a 30% increase in pond water consumption.

As shown in Figure 7.1, the flotation water system is isolated from the chemical plant water system. The pH of material in the tailings pond will be 6.5 or higher, based on laboratory tests.

The amount of phosphogypsum produced using concentrate from the carbonate flotation process is 3 percent less than phosphogypsum produced with untreated feed. This amounts to a reduction of 42,600 tons of gypsum per year for a 1,000 tpd P_2O_5 phosphoric acid plant equivalent to 0.13 tons of gypsum per ton of acid P_2O_5.

The projected DAP cost for using untreated rock is $190.05 per ton versus $182.99 per ton using the flotation concentrate. These data were generated by Jacobs DAPCOST model which is based on the rock characteristics and current prices of sulfur and ammonia. The major elements of savings consist of a 3 percent reduction in sulfuric acid consumption and a penalty charged to the untreated rock, because the MER of the resultant filter acid is 0.119, which is too high to make 18-46-0 DAP. The MER of the filter acid from concentrate is 0.097, which will allow DAP to be made from approximately 77 percent of the acid.

The DAPCOST model has been used by Jacobs for several years and the model inputs and outputs are given in Appendix E of this report.

8.2 Proposed Program

A Phase II program is recommended based on the results of the bench scale test program described in Sections 4.0, 5.0 and 6.0 and based on the potential advantages of the process as described in Section 8.1. The proposed Phase II program includes pilot scale beneficiation testing, pilot scale acidulation testing, and a technical/economic analysis to determine the overall impacts of the carbonate flotation module on phosphoric acid production cost.

8.2.1 Beneficiation Testing

The potential benefits of reducing the carbonate content of ground phosphoric acid reactor feed, as demonstrated by bench scale testing, warrant additional testing in a pilot scale flotation program. A continuous pilot plant more closely simulates the conditions in a commercial plant and accordingly developes more reliable data for evaluating the technical and economic potential of the process. A continuous pilot plant may reveal problems not readily evident from bench scale batch tests.

The proposed beneficiation testing program includes:

- anionic pilot scale rougher flotation of selected phosphate rock
    - to be examined at 2 to 3 levels for test parameters established from the bench scale test program
    - to be examined in a mechanical flotation cell and a column flotation cell
    - to examine the effects of recycle water use
- bench scale testing to support the pilot scale beneficiation tests.

8.2.2 Technical/Economic/Analysis

The potential benefits of adding a flotation module to an existing phosphoric acid plant to reduce the carbonate content of the rock may be offset by the capital and operating costs of the flotation module and the resultant P_2O_5 recovery loss to the carbonate tailings. The proposed Phase II beneficiation and acidulation testing will provide information to more accurately quantify the benefits and disadvantages of carbonate reduction on the overall production of DAP. Included in the technical/economic analysis are:

- an update of the Phase I estimated cash operating costs based on continuous pilot scale flotation test results
- an update of the Phase I estimated capital cost to construct a hypothetical flotation plant to remove carbonates from ground phosphoric acid plant reactor feed
- an estimate of the net effect of the process on phosphoric acid production, phosphogypsum production and disposal, and DAP production.

APPENDIX IS AVAILABLE IN FIPR LIBRARY