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EVALUATION OF DOLOMITE SEPARATION TECHNIQUES

Prepared By

Hassan El-Shall
Global Marketing and Consulting

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FIPR

Florida Institute of
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Research Directors

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Jinrong P. Zhang	**-Mining & Beneficiation**
Steven G. Richardson	**-Reclamation**
Gordon D. Nifong	**-Environmental Services**

Florida Institute of Phosphate Research
1855 West Main Street
Bartow, Florida 33830
(863) 534-7160
Fax:(863) 534-7165

EVALUATION OF DOLOMITE SEPARATION TECHNIQUES

Final Report
(FIPR Contract No. 93-02-094)

Submitted by:

Hassan El-Shall
Global Marketing and Consulting
2009 Paladin Ct.
Valrico, FL 33594

Prepared for:

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

Contract Manager: Jinrong (Patrick) Zhang

October 1994

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PERSPECTIVE

By Patrick Zhang
Director of Beneficiation Research

Among the deleterious impurities in phosphate rock used for producing phosphoric acid, dolomite is the most troublesome. As the carbonate proportion increases, the consumption of sulfuric acid in fertilizer manufacture also increases per ton of P_2O_5 produced. In addition, the carbonates contain a significant percentage of MgO in the form of dolomite and dolosilt. The MgO forms a gel that reduces the filtering capacity and ties up an equivalent portion of the P_2O_5 when acidulated.

With the depletion of the higher grade, easy-to-process Bone Valley deposits, the central Florida phosphate industry has been forced to move into the lower grade, more contaminated ore bodies from the Southern Extension. Although carbonaceous materials in an igneous phosphate deposit can be successfully removed by flotation methods, separation of dolomite from a sedimentary deposit using flotation techniques has not been commercially successful. The major dolomite problem in the future matrix will be with the pebble fraction.

Separation of dolomite from phosphate has been one of the most active research areas in phosphate mineral processing. As a result, many processes have been proposed: 1) direct flotation of phosphate with carbonate depressants; 2) reverse flotation of carbonate with phosphate depressants; 3) rapid change of conditioning parameters; 4) physical methods; 5) calcination; and 6) acid leaching.

In an effort to identify the most efficient, economical, and environmentally sound technique for processing Florida dolomitic ores, FIPR initiated an in-house research project to evaluate five flotation separation processes utilizing the same high dolomite pebble feed (FIPR #89-02-082S). This program was later contracted out to Global Marketing and Consulting (GMC), which included two more seemingly promising processes: a physical method (cycloning) and a selective flocculation scheme.

The evaluation results did not duplicate most of the reported data by the developers. This has been attributed, in some cases, to the difference in hardness of waters used by different researchers From the standpoint of overall metallurgical performance, the IMCF process stands alone.

It should be pointed out that all of the processes achieved dolomite removal by sacrificing the recovery substantially (30-60%). Even the "optimum" (IMCF) process recovered only about 60% of the P_2O_5 from the pebble feed. Therefore, none of the processes evaluated may be considered to be "ideal", and the quest for a feasible dolomite separation process is still valid.

TABLE OF CONTENTS

APPENDIX A

APPENDIX B

LIST OF TABLES

LIST OF TABLES (continued)

LIST OF FIGURES

EXECUTIVE SUMMARY

Several dolomite separation policies have been developed through FIPR's funded research including University of Florida(UF), University of Alabama(UA), and U.S. Bureau of Mines. Other processes have been developed by IMC and TVA. In an effort to identify the most efficient, economically viable, and environmentally sound process, FIPR's Board of Directors funded an in-house research project(FIPR # 89-02-82) to evaluate these five flotation separation processes utilizing similar feed material. Each investigator was to report the best conditions of his process that can be utilized to separate the dolomite impurities.

The first objective of this project was to test the reported optimum conditions for each process and compare the results with those previously reported.

Data from the in-house project indicated that grinding high MgO pebbles resulted in the concentration of MgO in the fines(-200 Mesh). Thus, the second objective of this project was to test the possibility of separating MgO impurities with the fine fraction utilizing gravity separation techniques such as hydrocyclones. Other techniques such as selective flocculation were to be explored for separating dolomite from the fine fractions.

The following is a summary of the results obtained in these studies:

A- Flotation Testing:

Laboratory comparison of five phosphate-dolomite-silica flotation separation processes, developed by various investigators, was completed using a 26 % P_2O_5/2+ % MgO Florida pebble phosphate supplied by IMCF as the test sample. The current test results were compared with the previous investigators' reported results obtained during 1993 using representative samples of the same pebble phosphate. The current tests were performed using the previously reported "optimum" test conditions with some variation in flotation reagent levels used when deemed necessary for pH adjustment, adequate froth maintenance, particle flotation, etc. Two to four flotation tests were performed for each process at or near the reported "optimum" flotation parameter levels.

Rodmill wet grinding was performed on multiple batches of the pebble to yield -28, -35, and -48 mesh products for desliming and flotation test work. Deslimed flotation feeds analyzed 26.0 - 26.4% P_2O_5, 15.4 - 17.5% Insol and 1.2 - 1.5+% MgO. Recoveries of P_2O_5 and MgO in the deslimed flotation feeds were 64 - 72% and 36 - 46%, respectively. Dry screen analyses performed on samples of each deslimed flotation feed showed virtually no tramp oversize particles were present and that about 3-5% fine "near size" particles were present.

A total of fifteen dolomite separation flotation tests were performed, and eleven complete material balances were calculated from the resultant data. The best overall processing performance was obtained using the IMCF cationic process. A brief description of each process effectiveness is summarized as follows:

(1) IMCF Process - Effective using either 28/150 mesh or 35/150 mesh flotation feed. Concentrates were readily produced analyzing 31.0-31.6% P_2O_5, 3.0-5.0 % Insol, and 0.74-0.84% MgO at 55-60% P_2O_5 recovery from the original pebble sample (80-90% P_2O_5 recovery from the dolomite separation stage feed). These results were in good agreement with the previously reported results.

(2) U.S.B.M. Process - Not as effective with 28/150 mesh attrition scrubbed feed as previously reported. Concentrates analyzed 30.3-31.7% P_2O_5, 3.1-4.4% Insol, and 1.22-1.45 MgO at 27-54% P_2O_5 recovery from the original pebble sample (46-72% P_2O_5 recovery from the flotation feed). Analytical results previously reported were concluded to be erroneous when material balance calculations were shown to be MgO deficient. Current testwork required considerably more flotation collector to obtain the highest reported P_2O_5 recovery.

(3) UF Process - Not as effective as indicated by previously reported testwork. The best concentrate obtained analyzed 31.5% P_2O_5, 2.9% Insol, and 1.04% MgO at 36% P_2O_5 recovery from the - original pebble sample (55% P_2O_5 recovery from the flotation feed). Sodium silicate was required to obtain selectivity in the initial flotation stage, and sulfuric acid requirement for pH control was significantly higher than previously reported. The process was very sensitive to pH changes and difficult to control during the second flotation stage. Current tests included a final silica flotation stage whereas the reported testwork omitted this processing step. Feed size processed was 35/150 mesh.

(4) UA Process - Reported to have failed to float dolomite or phosphate as designed when Bartow tap water was used during processing. Current testwork confirmed this reported result. Precipitation of fatty acid collector by water hardness ions was concluded to be partially responsible for the poor flotation response. Feed size processed was 35/150 mesh. Dolomite particles coarser than 48 to 65 mesh do not readily float using this process. Previously reported MgO analyses were concluded to be excessively high as presented in the I. Anazia report.

(5) TVA Process - Not as effective using 48/325 mesh attrition scrubbed feed as previously reported. Concentrates analyzed 30.7-30.8% P_2O_5, 3.0-3.8% Insol, and 1.40-1.51% MgO at 65-66% P_2O_5 recovery from the original pebble sample (96-97% P_2O_5 recovery from the scrubbed, deslimed flotation feed). A 150% increase in dolomite collector dosage failed to lower the phosphate concentrate MgO to 1.0% as previously reported. The scrubbed flotation feed was observed to produce some slime during attempted dolomite flotation. Partial precipitation of the fatty acid dolomite collector by water hardness ions was suspected to contribute to the failure of this process to effectively float dolomite.

Flotation reagent cost for the IMCF process ranged from $2.48 -$2.68 per ton of concentrate for the successful tests performed compared to 2.69-$2.86 per ton of concentrate previously reported. Flotation reagent costs for the other less effective processes ranged from about $1.75 to $4.25 per ton of inferior grade concentrate. Detailed reagent consumptions and costs, and cost of power consumption in grinding and scrubbing as required by each process are presented in the report text for comparison.

All tested flotation processes used biodegradable reagents. In other words, all processes are not expected to pollute the environment.

B-Cyclone Testing

Grinding high MgO samples (3.0% +) has produced approximately 35% -400mesh and about 64% of the total MgO was found in the -400 mesh size fraction. Screen analysis of ground rock coarser than 400 mesh indicated that part of the MgO was not preferentially ground and was consistent with the P_2O_5 content of the individual size fractions from 28 down to 400 mesh.

Cyclone tests conducted by Met Pro Supply, Inc. demonstrated. that about 88% of the MgO in the -400 mesh fines could be removed in the overflow or 57% of the total MgO present. This same overflow will contain approximately 27% of the available P_2O_5.

C-Selective Flocculation Testing

Selective flocculation tests conducted according to the procedure described in the progress report of FIPR # 89-02-083 failed to produce any selectivity. In other words, bulk flocculation of both dolomite and phosphate minerals was obtained. This was attributed to slime coating and inefficient dispersion. More extensive research is needed in this area.

INTRODUCTION

With the depletion of the higher grade, lower cost phosphate rock in the Bone Valley Member deposits, the Central Florida Phosphate Industry has been forced to move into the higher cost, lower quality deposits to the south.

Geologically, the Bone Valley deposits, because of reworking, were primarily contaminated with liberated silica that could easily be removed by conventional low cost flotation methods. The enrichment by replacing calcium carbonate with francolite apatite in the phosphate pellets was fairly complete with a relatively small percentage of free carbonate available. As the mining operations move to the south, this replacement is less complete causing an increase in the carbonate/phosphate ratio.

As the carbonate proportion increases, the consumption of sulphuric acid in fertilizer manufacture also increases per ton of P_2O_5 produced. In addition, the carbonates contain a significant percentage of MgO in the form of dolomite and dolosilts. The MgO forms a gel which reduces the filtering capacity and ties up an equivalent portion of the P_2O_5 when acidulated.

Research work to remove deleterious dolomite has tested gravity concentration, heavy media separation and froth flotation methods. Although there has been some success in dolomite removal using heavy media separation it still represents the major problem in rock quality from nearly all the major phosphate resources in the world.

Several dolomite separation processes have been developed through FIPR's funded research including University of Florida(UF), University of Alabama(UA), U.S. Bureau of Mines. Other processes have been developed by IMC and TVA. In an effort to identify the most efficient, economically viable, and environmentally sound process, FIPR's Board of Directors funded an in-house research project(FIPR # 89-02-82) to evaluate these five flotation separation processes utilizing similar feed material. Each investigator was to report the best conditions of his process that can be utilized to separate the dolomite impurities. The received reports are included in Appendix A.

The first objective of this project was to test the reported optimum conditions for each process and compare the results with those previously reported.

Data from the in-house project indicated that grinding high MgO pebbles resulted in the concentration of MgO in the fines(-200 Mesh). Thus, the second objective of this project was to test the possibility of separating MgO impurities with the fine fraction utilizing gravity separation techniques such as hydrocyclones. Other techniques such as selective flocculation were to be explored for separating dolomite from the fine fractions.

RESULTS AND DISCUSSION

l- Flotation Testing

1.1 Feed Sample Preparation and Characterization: The -3 mesh pebble phosphate sample used for all test work was obtained from IMCF's Four Corners mine in late 1992. This high-MgO washer "reject", in storage at FIPR, was coned and quartered to yield four fractions for analysis and processing. Analysis of the head sample obtained by riffling a quartered portion of the pebble showed the following chemical composition:

	%
P_2O_5	25.58
(BPL)	(55.90)
Insol	12.43
MgO	2.03
CaO	40.95

Three of the individual quartered pebble samples were riffled to yield appropriate weights (-1200 g per batch) for rod milling and desliming. Stage grinding at -60% solids in the laboratory batch rodmill was performed on the multiple batch samples to yield -28 mesh, -35 mesh and -48 mesh products for desliming. Material balances for the grinding and desliming products are presented in Table 1.

Flotation feed sizes used to evaluate the various processes to be tested were as follows:

Process	Feed Size, Tyler Mesh
IMCF	28/150 & 35/150
U.S.B.M.	28/150
UF	35/150
UA	35/150
T.V.A.	48/325

Dry screen analyses were performed on each of the three deslimed feed samples prepared from the rodmilled products. Results are summarized in Table 2.

Table 2 data shows that approximately 28-35% wt. of grinding slimes were produced representing a loss of 28-35% of the P_2O_5 values and a rejection of 50-64% of the MgO values present in the original pebble. The resultant deslimed flotation feed samples analyzed 1.2-1.4% MgO (by direct analysis) or 1.4-1.6% MgO (calculated arithmetic means from all flotation test products).

Table 1

SIZE ANALYSES OF DESLIMED FEEDS

TYLER MESH	% WT.	CUM. % WT.
28 M GRIND		
+28	Trace	Trace
28/35	31.4	31.4
35/48	25.2	56.6
48/65	19.3	75.9
65/100	12.1	88.0
100/150	8.6	96.6
-150	3.4	100.0
COMP.	100.0	-
35 M GRIND		
+35	0.8	0.8
35/48	30.3	31.1
48/65	30.8	61.9
65/100	19.3	81.2
100/150	13.2	94.4
-150	5.6	100.0
COMP.	100.0	-
48 M GRIND		
+48	Trace	Trace
48/65	30.5	30.5
65/100	24.0	54.4
100/150	16.5	71.0
150/200	13.9	84.9
200/325	9.5	94.4
-325	5.6	100.0
COMP.	100.0	-

Table 2

GRINDING/DESLIMING MATERIAL BALANCES

		ANALYSIS, %					% DIST.	
PRODUCT	% WT.	P_2O_5	INSOL	MgO	CaO	MgO/P_2O_5	P_2O_5	MgO
28/150 Mesh	71.7	26.42	15.40	1.39	40.11	0.053	71.9	44.7
- 150 Mesh	28.3	26.11	7.68	4.35	42.91	0.167	28.1	55.3
Total Pebble	100.0	(26.33)	(13.21)	(2.23)	(40.90)	0.085	100.0	100.0
Approximate Weight Processed = 25 lbs.								
35/150 Mesh	64.6	25.94	17.51	1.21	39.85	0.047	64.2	36.0
- 150 Mesh	35.4	26.43	7.73	3.93	42.75	0.149	35.8	64.0
Total Pebble	100.0	(26.12)	(14.05)	(2.17)	(40.87)	0.083	100.0	100.0
Approximate Weight Processed = 17 lbs.								
48/325 Mesh	70.5	26.38	16.27	1.27	41.25	0.048	70.7	38.6
- 325 Mesh	29.5	26.15	5.84	4.81	43.18	0.184	29.3	61.4
Total Pebble	100.0	(26.31)	(13.19)	(2.32)	(41.82)	0.088	100.0	100.0
Approximate Weight Processed = 7 lbs.								
28/150 Mesh*	71.7	26.19	16.22	1.58	40.11	0.060	71.8	47.9
- 150 Mesh	28.3	26.11	7.68	4.35	42.91	0.167	28.2	52.1
Total Pebble	100.0	(26.17)	(13.80)	(2.36)	(40.90)	0.090	100.0	100.0
35/150 Mesh*	64.6	25.97	16.56	1.49	39.85	0.057	64.2	40.9
- 150 Mesh	35.4	26.43	7.73	3.93	42.75	0.149	35.8	59.1
Total Pebble	100.0	(26.13)	(13.44)	(2.35)	(40.87)	0.090	100.0	100.0
48/325 Mesh*	70.5	26.18	16.83	1.39	41.25	0.038	70.7	40.8
- 325 Mesh	29.5	26.15	5.84	4.81	43.18	0.184	29.5	59.2
Total Pebble	100.0	(26.17)	(13.59)	(2.40)	(41.82)	0.092	100.0	100.0

* Calculated analyses from all flotation tests.

The Table 1 results illustrate the granulometry of each flotation feed sample. Misplaced "fines" ranged from 3.4% wt. in the 28/150 mesh sample to 5.6% wt. in the 35/150 mesh and the 48/325 mesh samples. Tramp oversize was insignificant in all three samples.

1.2 <u>Flotation Test Procedures and Results:</u> All laboratory flotation tests were performed using the 500 g Denver 'cell. Attrition scrubbing of flotation feed was performed using the Denver cell (<50% solids) or the Denver attritioning box attachment (>50% solids). Tap water was used for all feed preparation and flotation testwork. Reagents used previously by the various investigators at IMCF, U.S.B.M., UF, UA, and T.V.A. were used during the current investigation[1]. Feed scrubbing, conditioning and flotation times, % solids, pH, etc. were maintained as close to the previously tested levels as possible, and additional tests using variations in previously reported reagent levels were also performed when practical judgement indicated improved results might be obtained. Complete processing material balances were calculated, based upon flotation feed and based upon original pebble, for all relevant tests.

1.2.1. <u>IMCF Process.</u> The IMCF processing flowsheet is pictured in Figure 1[2]. In this process the silica (insol) is initially floated from phosphate using an amine condensate. The cell underflow is then dewatered, conditioned at high % solids at a slightly acid pH with tallow amine acetate plusdiesel fuel, and subjected to rougher/cleaner/re-cleaner flotation of phosphate from dolomite.

In past testwork, this process has been effective in removing dolomite from phosphate present in deslimed feed as coarse as 24 mesh. In the present test series, both 28/150 mesh and 35/150 mesh feeds were processed. A portion of each feed sample was divided into several -500 g batches (dry basis). Each batch of 28/150 mesh feed was subjected to cationic flotation, using Azamine 36A[3] plus diesel fuel at natural pH, to reject silica into the froth. The amine tails were cornposited weighed, dried and analyzed. The wet phosphatic concentrate was divided into several -500 g charges for dolomite separation testwork. The same procedure was used with the 35/100 mesh feed batches.

Two dolomite separation tests, using Armac T plus diesel fuel as the phosphate collector, were performed on both feed samples (amine concentrates from silica pre-floats). Complete material balances for these tests are presented in Tables 3&4 (28/150 mesh)

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[1] The one exception being dodecylamine hydrochloride - not used

[2] Copied from l/19/93 IMCF report.

[3] Pre-determined level derived from previous testwork.

and Tables 5&6 (35/150 mesh) using different reagent levels. Tables 7&8 present the results obtained, for comparisons, during previous (early 1993) tests performed by IMCF.

Examination and comparison of the Table 3-8 results reveals the effectiveness and relative reproducibility of the process to yield phosphate concentrates analyzing > 31% P_2O_5 and < 1.0% MgO at 80-90+ % P_2O_5 recovery from the dolomite separation stage feed (55-60% overall P_2O_5 recovery). Note that when excessive collector dosage was used (Table 4) the phosphate concentrate contained > 1.0% MgO.

FIGURE 1
Simplified Pebble Processing Flowsheet
IMCF CATIONIC PROCESS

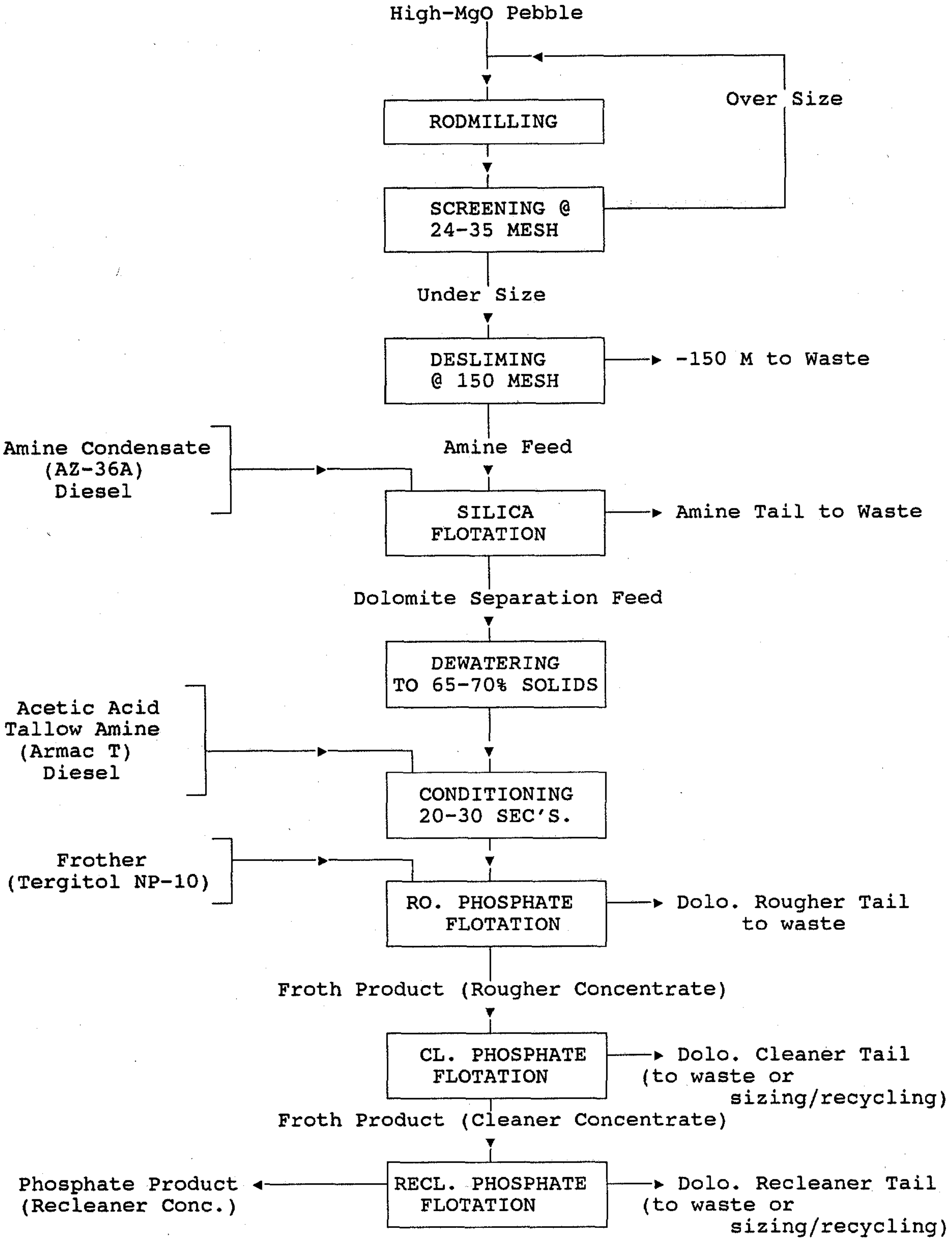

Table 3

IMCF Process Material Balance For 28/150M Feed - Test 1

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	46.8	31.08	5.01	0.81	46.26	55.5	17.5
Dolomite Re-Cleaner Tail.	2.7	27.62	4.24	3.76		2.8	4.6
Dolomite Cleaner Tail.	1.5	25.08	4.77	4.84		1.4	3.2
Dolomite RougherTail.	10.8	25.09	4.00	5.16		10.3	25.8
Silica Amine Tail.	9.9	4.65	85.41	0.23		1.8	0.9
-150 M Rodmill Slime	28.3	26.11	7.68	3.67		28.2	48.0
Head Composite	100.0	26.33	13.58	2.17		100.0	100.0
Phosphate Cleaner Conc.	49.5	30.90	4.98	0.97		58.3	22.1
Dolomite Separation Feed	61.8	29.74	4.77	1.79		70.0	51.1
Silica Flotation Feed	71.7	26.28	15.91	1.57		71.8	52.0

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	75.8	31.08	5.01	0.81	46.26	79.2	34.1
Dolomite Re-Cleaner Tail.	4.4	27.62	4.24	3.76		4.1	8.9
Dolomite Cleaner Tail.	2.4	25.08	4.77	4.84		2.0	6.7
Dolomite Rougher Tail.	17.4	25.02	4.00	5.16		14.7	50.3
Dolomite Separation Feed	100.0	29.20	4.84	1.79		100.0	100.0
Phosphate Cleaner Conc.	80.2	30.17	4.98	0.97		83.4	43.0

REAGENTS	Lb/T. Feed
Azamine A-36A	1.20
Diesel Fuel	0.60
Acetic Acid	0.60
Armac T.	1.38
Diesel Fuel	4.14
Tergitol NP-10	0.02

Table 4

IMCF Process Material Balance For 28/150M Feed - Test 2

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	53.0	31.13	4.82	1.12	46.57	62.2	30.0
Dolomite Re-Cleaner Tail.	1.6	21.59	4.70	6.85		1.3	5.2
Dolomite Cleaner Tail.	1.4	22.92	4.12	6.07		1.2	3.8
Dolomite Rougher Tail.	5.8	25.96	4.01	4.65		5.7	12.8
Silica Amine Tail.	9.9	4.65	85.41	0.23		1.7	0.9
- 150 M Rodmill Slime	28.3	26.11	7.68	3.67		27.9	47.3
Head Composite	100.0	26.52	13.55	2.11		100.0	100.0
Phosphate Cleaner Conc.	54.6	30.86	4.82	1.28		63.5	35.2
Dolomite Separation Feed	61.8	30.21	4.72	1.70		70.4	51.8
Silica Flotation Feed	71.7	26.68	15.87	1.49		72.1	52.7

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	85.7	31.13	4.82	1.12	46.57	88.3	55.8
Dolomite Re-Cleaner Tail.	2.6	21.59	4.70	6.85		1.9	10.5
Dolomite Cleaner Tail.	2.3	22.92	4.12	6.07		1.7	8.1
Dolomite Rougher Tail.	9.4	25.96	4.01	4.65		8.1	25.6
Dolomite Separation Feed	100.0	30.21	4.72	1.72		100.0	100.0
Phosphate Cleaner Conc.	88.3	30.85	4.81	1.29		90.2	66.3

REAGENTS	Lb/T. Feed
Azamine A-36A	1.20
Diesel Fuel	0.60
Acetic Acid	0.60
Armac T	1.72
Diesel Fuel	5.17
Tergitol NP-10	0.03

Table 5

IMCF Process Material Balance for 35/150M Feed - Test 3

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	48.4	31.66	3.98	0.84	46.19	58.4	17.9
Dolomite Re-Cleaner Tail.	2.5	25.84	3.50	4.65		2.5	5.2
Dolomite Cleaner Tail.	1.7	18.91	3.69	10.79		1.2	7.9
Dolomite Rougher Tail.	2.3	12.74	4.01	14.25		1.1	14.4
Silica Amine Tail.	9.7	2.97	90.08	0.16		1.1	0.4
- 150 M Rodmill Slime	35.4	26.43	7.73	3.50		35.7	54.2
Head Composite	100.0	26.23	13.65	2.29		100.0	100.0
Phosphate Cleaner Conc.	50.9	31.37	3.96	1.04		60.9	23.1
Dolomite Separation Feed	54.9	30.21	3.95	1.89		63.6	45.4
Silica Flotation Feed	64.6	26.11	16.89	1.62	64.3	45.8	

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	88.1	31.66	3.98	0.84	46.19	92.3	39.4
Dolomite Re-Cleaner Tail.	4.6	25.84	3.50	4.65		4.0	11.2
Dolomite Cleaner Tail.	3.2	18.91	3.69	10.79		2.0	18.6
Dolomite Rougher Tail.	4.1	12.74	4.01	14.25		1.7	30.8
Dolomite Separation Feed	100.0	30.21	3.95	1.88		100.0	100.0
Phosphate Cleaner Conc.	92.7	31.37	3.96	1.03		96.3	50.6

REAGENTS	Lb/T. Feed
Azamine A-36A	1.40
Diesel Fuel	0.70
Acetic Acid	0.60
Armac T	1.53
Diesel Fuel	4.59
Tergitol NP-10	0.03

Table 6

IMCF Process Material Balance For 35/150M Feed - Test 4

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	46.8	31.43	2.95	0.74	46.73	56.4	15.9
Dolomite Re-Cleaner Tail.	3.5	27.26	3.05	3.21		3.6	5.0
Dolomite CleanerTail.	2.1	21.00	3.46	8.18		1.7	7.7
Dolomite RougherTail.	2.5	13.77	3.93	12.87		1.3	14.5
Silica Amine Tail.	9.7	2.97	90.08	0.16		1.1	0.5
-150 M Rodmill Slime	35.4	26.43	7.73	3.50		35.9	56.4
Head Composite	100.0	26.09	13.14	2.20		100.0	100.0
Phosphate Cleaner Conc.	50.3	31.13	2.96	0.91		60.0	20.9
Dolomite Separation Feed	54.9	29.95	3.02	1.73		63.0	43.1
Silica Flotation Feed	64.6	25.90	16.10	1.49		64.1	43.6

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	85.3	31.43	2.95	0.74	46.73	89.5	36.4
Dolomite Re-Cleaner Tail.	6.3	27.26	3.05	3.21		5.7	11.6
Dolomite Cleaner Tail.	3.8	21.00	3.46	8.18		2.7	17.9
Dolomite Rougher Tail.	4.6	13.77	3.93	12.87		2.1	34.1
Dolomite Separation Feed	100.0	29.96	3.02	1.73		100.0	100.0
Phosphate Cleaner Conc.	91.6	31.15	2.96	0.91		95.2	48.0

REAGENTS	Lb/T. Feed
Azamine A-36A	1.40
Diesel Fuel	0.70
Acetic Acid	0.60
Armac T	1.36
Diesel Fuel	4.08
Tergitol NP-10	0.03

Table 7

IMCF Process Material Balance For 28/150M Feed - Report of Previous Work

Product	% Wt.	P_2O_5	Insol	MgO	CaO	% Distribution P_2O_5	% Distribution MgO
		Analysis, %					
Phosphate Re-Cleaner Conc.	52.1	31.03	5.45	0.86		61.9	21.8
Dolomite Re-Cleaner Tail.	4.2	28.54	3.90	2.58		4.6	5.4
Dolomite Cleaner Tail.	3.5	24.78	4.12	4.54		3.3	7.7
Dolomite Rougher Tail.	5.5	20.19	4.56	7.40		4.2	19.9
Silica Amine Tail.	8.9	2.50	90.62	0.16		0.9	0.5
-150 M Rodmill Slime	25.8	25.46	6.55	3.56		25.1	44.7
Head Composite	100.0	26.13	13.16	2.06		100.0	100.0
Phosphate Cleaner Conc.	56.4	30.83	5.33	0.99		66.5	27.2
Dolomite Separation Feed	65.3	29.62	5.21	1.73		74.0	54.8
Silica Flotation Feed	74.2	26.36	15.46	1.54		74.9	55.3

Product	% Wt.	P_2O_5	Insol	MgO	CaO	% Distribution P_2O_5	% Distribution MgO
		Analysis, %					
Phosphate Re-Cleaner Conc.	79.8	31.03	5.45	0.86		83.6	40.1
Dolomite Re-Cleaner Tail.	6.5	28.54	3.90	2.58		6.2	9.9
Dolomite Cleaner Tail.	5.3	24.78	4.12	4.54		4.4	14.0
Dolomite Rougher Tail.	8.4	20.19	4.56	7.40		5.8	36.0
Dolomite Separation Feed	100.0	29.62	5.20	1.72		100.0	100.0
Phosphate Cleaner Conc.	86.3	30.83	5.33	0.99		89.8	50.0

REAGENTS	Lb/T. Feed
Azamine A-36A	1.10
Diesel Fuel	0.60
Acetic Acid	0.79
Armac T	1.58
Diesel Fuel	4.75
Tergitol NP-10	0.12

Table 8

IMCF Process Material Balance For 35/150M Feed - Report of Previous Work

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Re-Cleaner Conc.	47.5	31.63	3.02	0.77		57.9	18.7
Dolomite Re-Cleaner Tail.	2.9	28.39	2.48	2.56		3.2	3.5
Dolomite Cleaner Tail.	2.2	23.29	2.89	5.35		2.0	6.1

1.2.2 U.S.B.M. Process. The U.S.B.M. processing flowsheet is presented in Figure 2[4]. In this process the 28/150 mesh feed is vigorously attrition scrubbed (with NaOH added) for 20 minutes at 50% solids to reduce much of the softer dolomite to slime size, and the scrubbed sample is deslimed at 150 mesh. The scrubbed, deslimed feed is conditioned at -70% solids for 5 minutes with NaOH, oleic acid and fuel oil at pH = 9.0 - 9.2. The reagentized feed is subjected to rougher-cleaner flotation to float phosphate from silica and dolomite. Sodium silicate is added during cleaner flotation to aid depression of silica and dolomite.

Three flotation tests were performed using this process with three different collector levels. Two different scrubbing rpm settings were used. At 1100 rpm, too much scrubber slime was produced; consequently the final two tests were performed using 900 rpm to reduce the slime generated to a level closer to that previously obtained by U.S.B.M. investigators. Complete material balances for these tests are presented in Tables 9-11. Table 12 presents the comparative results obtained previously (mid 1993) by the U.S.B.M.

Examination and comparison of the Table 9-12 results illustrates the current and past performance of the process. It is obvious from the discrepancy in head sample analyses reported by the past and the present investigators that there is an analytical problem present. Tables 9-11 results show calculated head analyses of 25.8 - 26.3% P_2O_5, 13.4 - 14.4% Insol, and 2.15 - 2.30% MgO. The Table 12 (U.S.B.M.) results show calculated head analyses of 23.6% P_2O_5, 16.6% SiO_2, and 1.78% MgO. The previously issued U.S.B.M. report claimed feed analyses of 24.1% P_2O_5, 12.6% SiO_2, and only 0.82% MgO. Current calculations showed this flotation feed contained 24.9% P_2O_5, 20.9% SiO_2, and 1.12% MgO.

The U.S.B.M. reported optimum fatty acid[5] plus fuel oil collector level to be 2.5 lbs/ton of flotation feed. The current tests summarized in Tables 9-11 used 2.5, 3.75 and 5.0 lbs. oleic acid plus fuel oil collector, respectively, per ton of flotation feed. Phosphate concentrates analyzed 31.7 - 30.3% P_2O_5, 3.1 - 4.4% Insol, and 1.22 - 1.54% MgO at 45.6 - 72.3% recovery of P_2O_5 from the flotation feed using the two lowest collector levels. Overall P_2O_5 *recovery* was 27.4 - 49.1 % for these tests . Using the highest collector level (2 x U.S.B.M. quantity), the phosphate concentrate analyzed 29.6% P_2O_5, 5.2% Insol, and 1.66% MgO at 79.4% recovery of P_2O_5 from the flotation feed (53.6% overall P_2O_5 recovery). The current flotation testwork showed that twice the previously reported collector level was required to produce an equivalent

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[4] Copied from 6/15/93 U.S.B.M. report.

[5] Reported as "a typical fatty acid".

recovery of P_2O_5 at an equal concentrate % P_2O_5. However, the current concentrate analyzed > 1.6% MgO and not the 1.1% MgO reported by U.S.B.M.

FIGURE 2
Simplified Pebble Processing Flowsheet
U.S.B.M. Process - Proposed Flowsheet

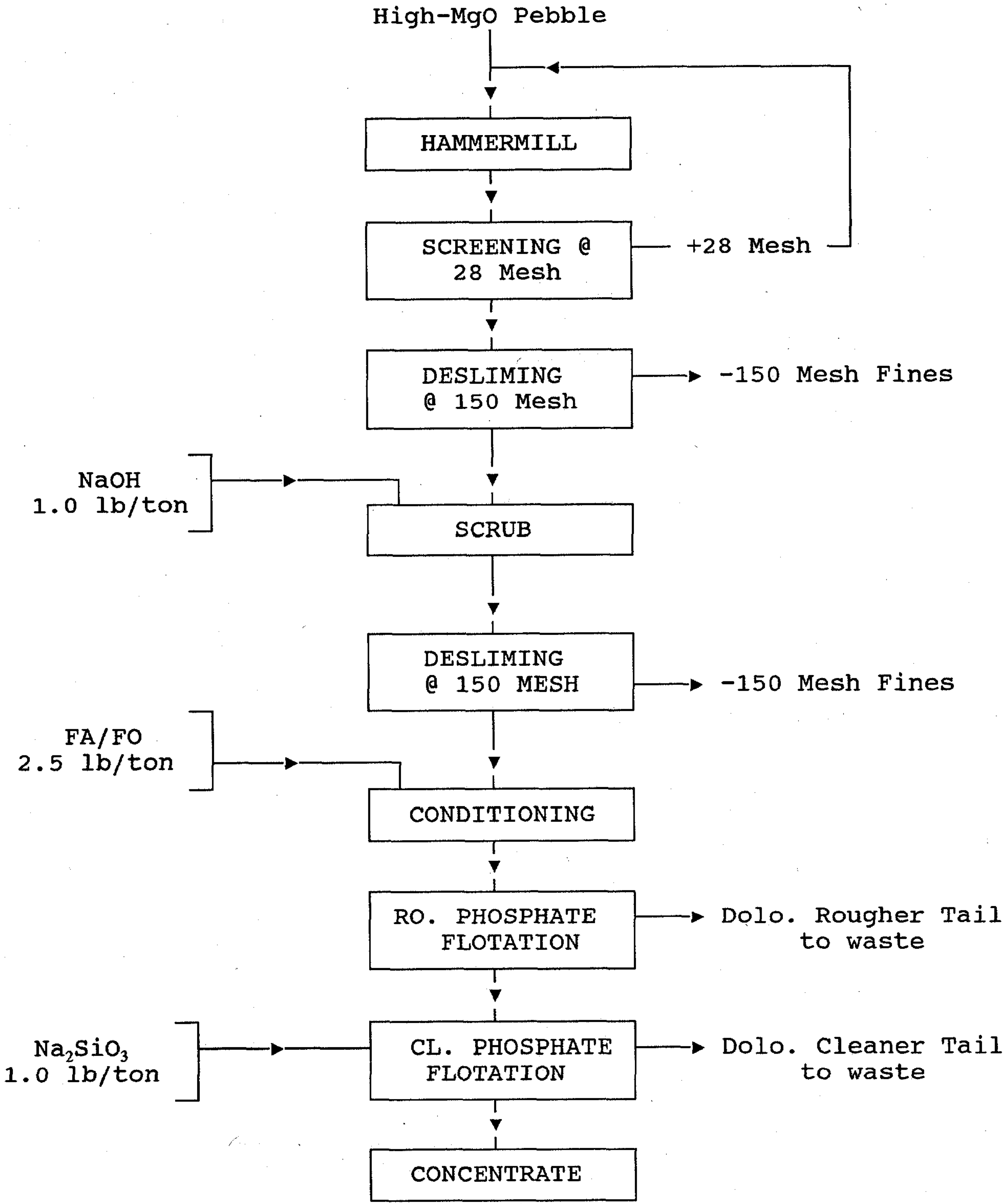

Table 9

U.S.B.M. Process Material Balance For 28/150M Feed - Test 2

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Cleaner Conc.	22.7	31.73	3.10	1.22	46.71	27.4	12.2
Dolomite Cleaner Tail.	3.9	29.22	6.20	1.71		4.3	3.0
Dolo./Silica Rougher Tail.	33.4	22.30	28.66	1.53		28.4	22.2
-150M Scrubber Slime	11.7	26.45	6.00	3.47		11.8	17.4
-150 M Rodmill Slime	28.3	26.11	7.68	3.67		28.1	45.2
Head Composite	100.0	26.27	13.38	2.30		100.0	100.0
Flotation Feed	60.0	26.32	17.52	1.43		60.1	37.4

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Cleaner Conc.	37.8	31.73	3.10	1.22	46.71	45.6	32.8
Dolomite Cleaner Tail.	6.5	29.22	6.20	1.71		7.2	7.6
Dolo./Silica Rougher Tail.	55.7	22.30	28.66	1.53		47.2	59.6
Flotation Feed	100.0	26.32	17.53	1.42		100.0	100.0

REAGENTS	Lb/T. Feed
Sodium Hydroxide	1.40
Oleic Acid	1.00
IPC Fuel Oil	1.50
"N' Sodium Silicate	1.00

Table 10

U.S.B.M. Process Material Balance For 28/150M Feed - Test 3

Product	% Wt.	Analysis, % P_2O_5	Insol	MgO	CaO	% Distribution P_2O_5	MgO
Phosphate Cleaner Conc.	42.2	30.31	4.37	1.54	45.79	49.1	30.0
Dolomite Cleaner Tail.	2.7	25.89	12.55	2.36		2.7	2.8
Dolo/Silica Rougher Tail.	22.9	18.29	41.16	1.15		16.1	12.0
-150 M Scrubber Slime	3.9	25.33	6.91	4.04		3.8	7.3
-150 M Rodmill Slime	28.3	26.11	7.68	3.67		28.3	47.9
Head Composite	100.0	26.06	14.05	2.17		100.0	100.0
Flotation Feed	67.8	26.08	17.12	1.44		67.9	44.8

Product	% Wt.	Analysis, % P_2O_5	Insol	MgO	CaO	% Distribution P_2O_5	MgO
Phosphate Cleaner Conc.	62.2	30.31	4.37	1.54	45.79	72.3	66.4
Dolomite Cleaner Tail.	4.0	25.89	12.55	2.36		4.0	6.6
Dolo/Silica Rougher Tail.	33.8	18.29	41.16	1.15		23.7	27.0
Flotation Feed	100.0	26.07	17.13	1.45		100.0	100.0

REAGENTS	Lb/T. Feed
Sodium Hydroxide	1.50
Oleic Acid	1.50
IPC Fuel Oil	2.25
"N' Sodium Silicate	1.00

Table 11

U.S.B.M. Process Material Balance for 28/150M Feed - Test 4

Product	% Wt.	Analysis, % P_2O_5	Insol	MgO	CaO	% Distribution P_2O_5	MgO
Phosphate Cleaner Conc.	46.6	29.62	5.22	1.66	45.58	53.6	36.0
Dolomite Cleaner Tail.	1.5	23.53	18.89	2.19		1.3	1.4
Dolo./Silica Rougher Tail.	19.7	16.41	46.89	0.82		12.5	7.5
-150 M Scrubber Slime	3.9	25.27	8.19	3.67		3.8	6.5
-150 M Rodmill Slime	28.3	26.11	7.68	3.67		28.8	48.6
Head Composite	100.0	25.76	14.44	2.15		100.0	100.0
Flotation Feed	67.8	25.63	17.63	1.42		67.4	44.9

Product	% Wt.	Analysis, % P_2O_5	Insol	MgO	CaO	% Distribution P_2O_5	MgO
Phosphate Cleaner Conc.	68.7	29.62	5.22	1.66	45.58	79.4	80.0
Dolomite Cleaner Tail.	2.2	23.53	18.89	2.19		2.0	3.0
Dolo./Silica Rougher Tail.	29.1	16.41	46.89	0.82		18.6	17.0
Flotation Feed	100.0	25.64	17.65	1.43		100.0	100.0

REAGENTS	Lb/T. Feed
Sodium Hydroxide	1.64
Oleic Acid	2.00
IPC Fuel Oil	3.00
"N' Sodium Silicate	1.00

Table 12

U.S.B.M. Process Material Balance for 28/150M Feed
Report of Previous Work

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Cleaner Conc.	43.1	29.60	7.20	1.10	54.00	54.0	26.8
Dolomite Cleaner Tail.	5.0	21.60	22.00	2.55		4.6	7.2
Dolo./Silica Rougher Tail.	17.1	14.00	55.20	0.75		10.1	7.2
-150 M Scrubber Slime	5.1	24.80	9.80	3.26		5.3	9.4
- 150 M Rodmill Slime	29.7	20.70	8.30	2.95		26.0	49.4
Head Composite	100.0	23.60	16.60	1.78		100.0	100.0
Flotation Feed	65.2	24.90	20.90	1.12		68.7	41.2

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Cleaner Conc.	66.1	29.60	7.20	1.10	54.00	78.6	65.0
Dolomite Cleaner Tail.	7.7	21.60	22.00	2.55		6.7	17.5
Dolo./Silica Rougher Tail.	26.2	14.00	55.20	0.75		14.7	17.5
Flotation Feed	100.0	24.90	20.90	1.12		100.0	100.0

* Reported as % SiO2

REAGENTS	Lb/T. Feed
Sodium Hydroxide	1.00
Oleic Acid	1.00
IPC Fuel Oil	1.50
"N' Sodium Silicate	1.00

1.2.3. UF Process. The UF processing flowsheet is illustrated in Figure 3[6]. This process was designed to beneficiate 35/150 mesh rodmilled, deslimed pebble, however, most of the testwork by the UF investigators was performed on only the 65/150 mesh feed fraction. The process consists of high % solids conditioning of the deslimed feed for 2.5 minutes at pH = 10 using Westvaco M-28 B tall oil and (optional) fuel oil as the phosphate collector followed by flotation of phosphate along with dolomite and fine silica. The rougher concentrate is reconditioned for about 30 seconds at 35 - 40% solids with sulfuric acid addition to maintain a 4.0 - 5.0 pH. Additional tall oil is added if deemed necessary, and the dolomite is floated as the slurry pH is maintained below -5.5 by sulfuric acid addition. Frother addition was used for froth control. The final processing step, although not performed by the UF investigators, consists of cationic flotation of the fine silica from the dewatered dolomite flotation cell underflow to yield a final phosphate concentrate and a siliceous amine tailing.

Three flotation tests were performed to evaluate this processing technique using 5.5 lb. M-28 B tall oil in the initial conditioning stage, and three different sulfuric acid levels during the selective de-oiling/dolomite flotation stage. The final cationic silica flotation stage was also performed during each test. The material balance flowsheets for the two most effective laboratory tests currently performed are presented in Tables 13 and 14. No material balance was prepared for the UF investigators test effort due to the lack of all needed data in their 8/93 report, however, the following single test data was found in the text of their report:

	Analyses, %		Distribution, %
Product	P_2O_5	MgO	P_2O_5
Phosphate Conc.	24.9	1.10	50.0
35/150 Mesh Feed	-	1.47	(100.0)

The data in Table 13 show that a phosphate concentrate was obtained analyzing only 28.2% P_2O_5, 11.8% Insol and 0.97% MgO at 43.4% recovery of P_2O_5 from the flotation feed (27.7% overall P_2O_5 recovery). These poor results were caused by the low recovery of the coarser phosphate particles and the excessive recovery of fine silica in the rougher flotation stage. Table 14 presents the data for a follow-up test using -0.4 lb. of sodium silicate per ton of feed for silica depression during rougher conditioning. The phosphate concentrate was improved and analyzed 31.5% P_2O_5, 2.9%

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[6] Copied from 8/93 University of Florida report.

Insol and 1.04% MgO at 55.4% recovery of P_2O_5 from the flotation feed (35.7% overall P_2O_5 recovery). The dolomite flotation stage and the cationic silica flotation stage were both very troublesome to perform. These flotation stages never really reached completion. Residual tall oil reagent appeared to be present on the cell underflow that caused a slow, continuing skin float to occur after the bulk of the float products were collected.

FIGURE 3
Simplified Pebble Processing Flowsheet
UF Two Stage Process - Proposed Flowsheet

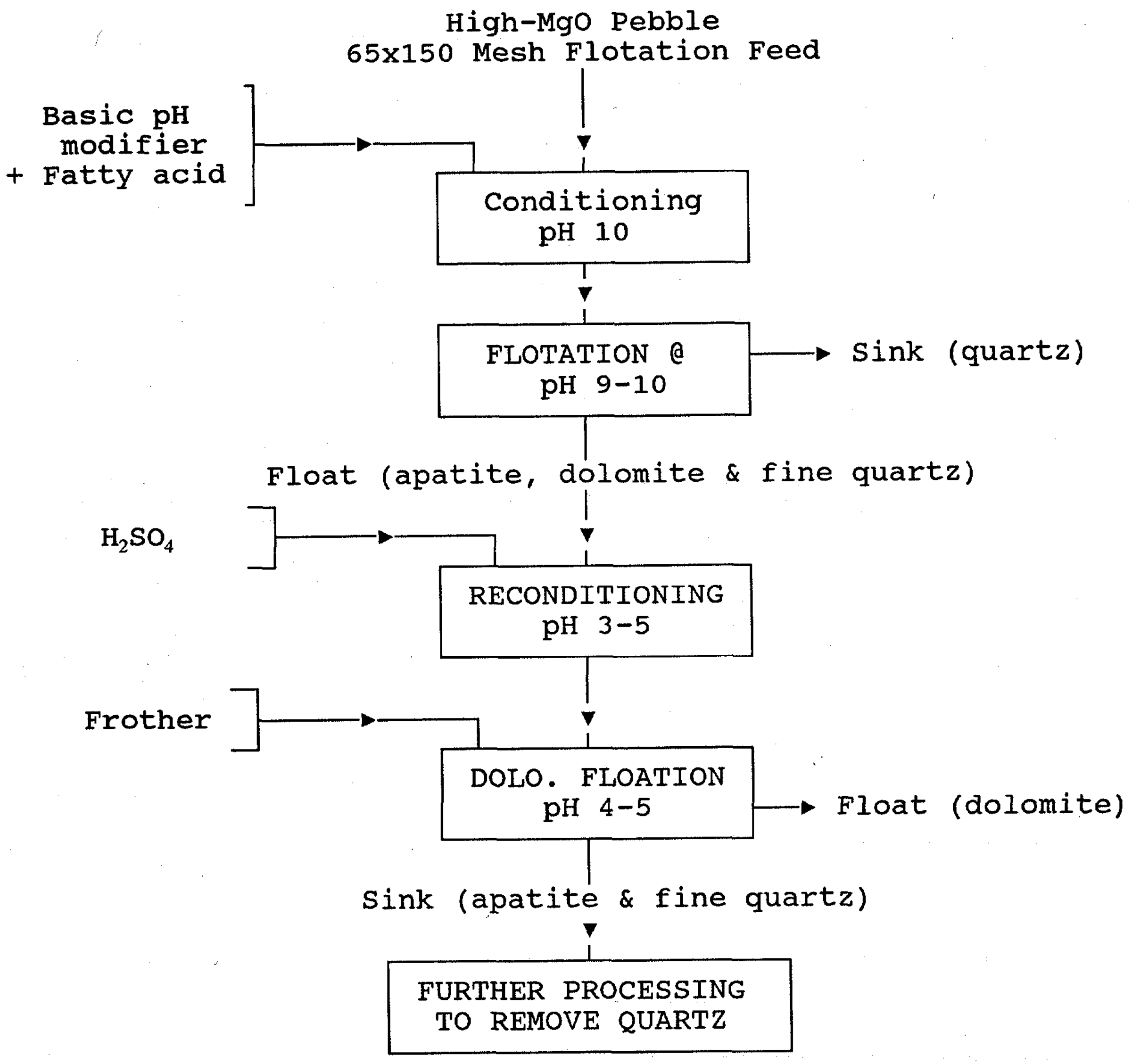

Table 13

UF Process Material Balance For 35/150M Feed - Test 2

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	25.5	28.17	11.82	0.97	42.44	27.7	11.6
Silica Amine Tail.	9.0	13.85	55.10	0.62		4.8	2.3
Dolomite Tail.	3.2	22.93	2.91	6.12		2.8	9.3
Rougher Tail.	26.9	27.55	9.74	1.51		28.6	19.1
-150 M Rodmill Slime	35.4	26.43	7.73	3.50		36.1	57.7
Head Composite	100.0	25.93	13.42	2.15		100.0	100.0
Flotation Feed	64.6	25.65	16.53	1.41		63.9	42.3

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	39.5	28.17	11.82	0.97	42.44	43.4	26.9
Silica Amine Tail.	13.9	13.85	55.10	0.62		7.5	6.4
Dolomite Tail.	5.0	22.93	2.91	6.12		4.5	22.0
Rougher Tail.	41.6	27.55	9.74	1.51		44.6	44.7
Flotation Feed	100.0	25.67	16.53	1.41		100.0	100.0
Amine Flot. Feed	53.4	24.46	23.09	0.88		50.9	33.3

REAGENTS	Lb/T. Feed
Sodium Hydroxide	2.10
M2-28B Tall Oil	5.50
IPC Fuel Oil	0.00
Sulfuric Acid	4.00
Aerofroth 65	0.00
Azamine A36-A	0.60
"N' Sodium Silicate	0.00

Table 14

UF Process Material Balance For 35/150M Feed - Test 3

Product	Analysis, %					% Distribution	
	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	29.8	31.49	2.88	1.04	46.89	35.7	14.3
Silica Amine Tail.	5.4	25.69	16.05	1.68		5.3	4.2
Dolomite Tail.	6.0	26.07	2.11	4.20		5.9	11.6
RougherTail.	23.4	19.66	38.20	1.16		17.5	12.5
-150 M Rodmill Slime	35.4	26.43	7.73	3.50		35.6	57.4
Head Composite	100.0	26.29	13.54	2.16		100.0	100.0
Flotation Feed	64.6	26.21	16.72	1.43		64.4	42.6

Product	Analysis, %					% Distribution	
	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	46.1	31.49	2.88	1.04	46.89	55.4	33.5
Silica Amine Tail.	8.4	25.69	16.05	1.68		8.2	9.8
Dolomite Tail.	9.3	26.07	2.11	4.20		9.2	27.3
Rougher Tail.	36.2	19.66	38.20	1.16		27.2	29.4
Flotation Feed	100.0	26.22	16.71	1.43		100.0	100.0
Amine Flot. Feed	54.5	30.95	4.92	1.14		63.6	43.3

REAGENTS	Lb/T. Feed
Sodium Hydroxide	2.10
M-28B Tall Oil	5.50
IPC Fuel Oil	1.10
Sulfuric Acid	5.80
Aerofroth 65	0.30
Azamine A36-A	0.50
"N' Sodium Silicate	0.40

1.2.4. UA Process. A flowsheet was not available from the UA report. Nevertheless, Figure 4 presents a flowsheet of the UA process constructed adopted from their previous reports to FIPR. This process was reported to have failed to float dolomite in the initial flotation step and phosphate in the second flotation step. The probable causes for the failure of this flowsheet were (1) water hardness (Ca^{+2} and Mg^{+2}) precipitating the fatty acid collector, and (2) dolomite particles too coarse to float with physically adsorbed fatty acid collector. A test performed, in this project, has confirmed these results.

The flowsheet in Figure 4 shows that the 35/150 mesh feed is scrubbed at 35% solids for 10 minutes and deslimed at 325 mesh. The deslimed feed is subjected to conditioning at 16% solids in a laboratory flotation machine at pH = 5.5 - 6.0 with a fatty acid to which was added small quantities of pine oil frother plus sodium hydroxide in order to float dolomite from phosphate and silica[7]. The flotation cell underflow is then further conditioned with sodium silicate, then with more fatty acid, and the phosphate is floated from the silica as the pH increases to 6.0 - 7.0.

A follow-up test was performed substituting a sulfonated oleic acid (sodium salt), labelled Tennessee Corp. 0A-5, as the dolomite flotation collector. Some dolomite flotation was achieved, however, the cell underflow product contained -1.2% MgO and further processing was abandoned. This exploratory test produced the following partial material balance:

Product	% wt.	Analysis, %			Distribution, %	
		P_2O_5	Insol	MgO	P_2O_5	MgO
Cell Underflow	87.0	26.20	18.27	1.19	87.1	65.0
Dolomite Froth	9.8	26.23	4.64	4.21	9.8	25.6
Scrubber Slime	3.2	25.10	4.32	4.63	3.1	9.4
Head Composite	100.0	(26.16)	(16.48)	(1.60)	100.0	100.0

This test only illustrates that when the sulfonated oleic acid reagent (not readily precipitated by water hardness ions) is used as the dolomite collector, some of the dolomite can be floated with a stable froth without using pine oil. This reagent is the basis of IMC's 1982 U.S. Patent 4,364,824.

The results presented in the UA report were believed to contain erroneous MgO analyses that were too high to be meaningful. The reported head sample analysis of 2.7% MgO was estimated to be 0.5 - 0.7% MgO higher than expected, and the 1.9% MgO reported for the 35/150 mesh flotation feed is also too high to be considered reliable.

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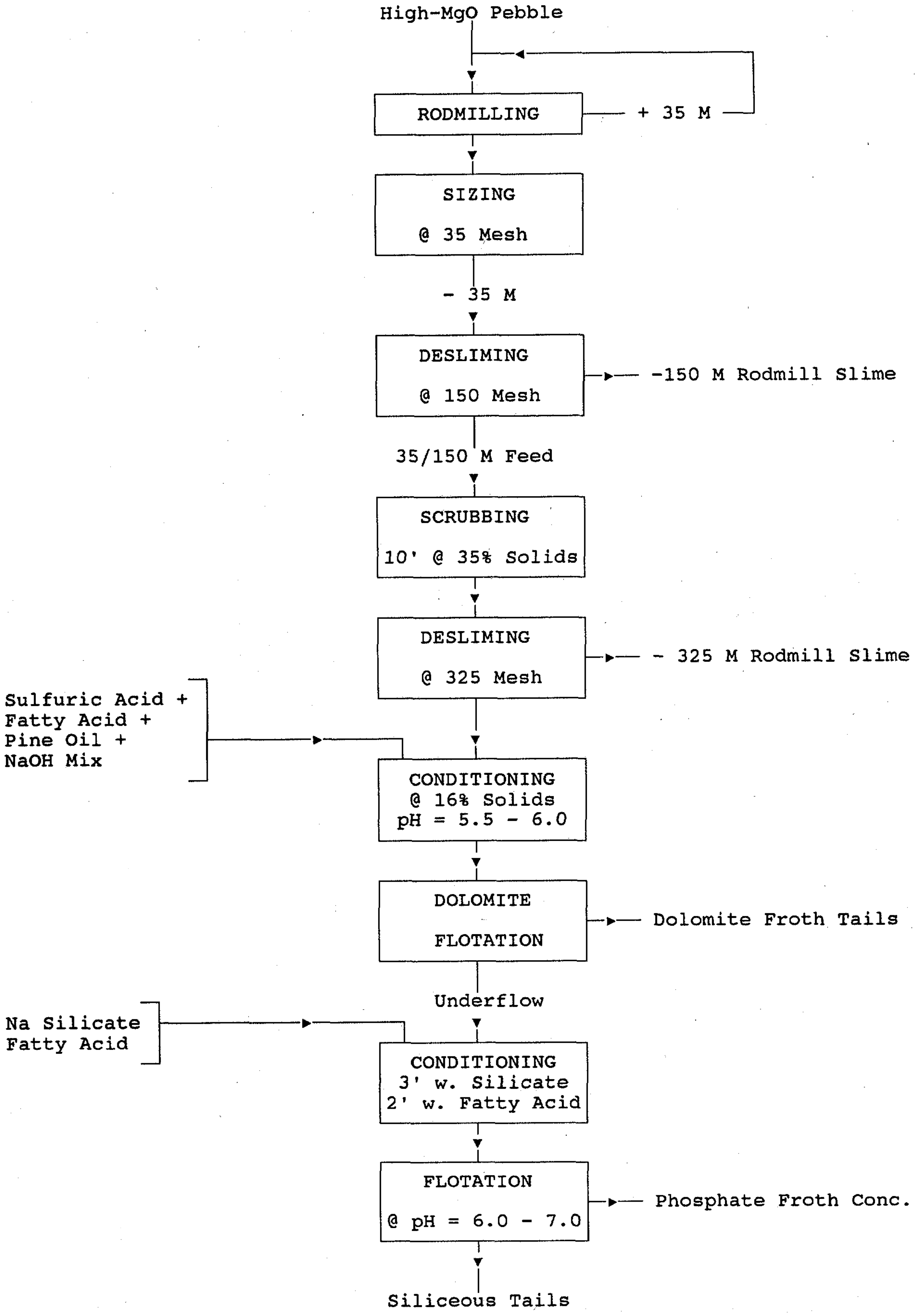
[7] stage addition of collector mixture was employed.

FIGURE 4
UA - Simplified Pebble Processing Flowsheet



1.2.5. T.V.A. Process. The T.V.A. processing flowsheet is shown in Figure 5[8]. This flowsheet utilizes rodmilling to -48 mesh followed by desliming at 400 mesh. The 48/400 mesh feed is attrition scrubbed for 5 minutes at 50% solids and deslimed again at 400 mesh in order to produce the dolomite flotation feed. The thoroughly deslimed feed is conditioned at 65% solids with diphosphonic acid, then with an oleic acid/pine oil mixture, and the dolomite is floated from phosphate and silica. Finally, cationic flotation with an appropriate amine collector is used to float the silica from phosphate.

Two flotation tests were performed using this process with two different levels of oleic acid/pine oil mixture. Complete material balances for these tests are presented in Tables 15 and 16. Table 17 presents the results reported by T.V.A. for comparison purposes.

The results in Tables 15 - 16 show that current tests yielded phosphate concentrates analyzing 30.7 - 30.8% P_2O_5, 3.0 - 3.8% Insol and 1.40 - 1.51% MgO at 96.6 - 97.5% recovery of P_2O_5 from the scrubbed, deslimed feed (64.9 - 66.0% overall P_2O_5 recovery). The data in Table 17 show that T.V.A. produced a phosphate concentrate analyzing 30.7% P_2O_5, 3.2% Insol and 1.01% MgO at 92.7% recovery of P_2O_5 from the scrubbed deslimed flotation feed (63.9% overall P_2O_5 recovery). The current testwork showed that the flotation cell pulp contained 0.6 - 0.8% wt. of -325 mesh slime produced during the dolomite flotation stage. The current testwork also utilized desliming at 325 mesh throughout the processing for simplicity in product handling. The current testwork failed to yield a phosphate concentrate analyzing as low as 1.0% MgO even when the oleic acid/pine oil collector level was increase to 150% of the level used by T.V.A.

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[8] Copied from 5/17/93 T.V.A. report.

FIGURE 5
Simplified Pebble Processing Flowsheet
TVA's Diphophonic Depressant Process - Proposed Flowsheet

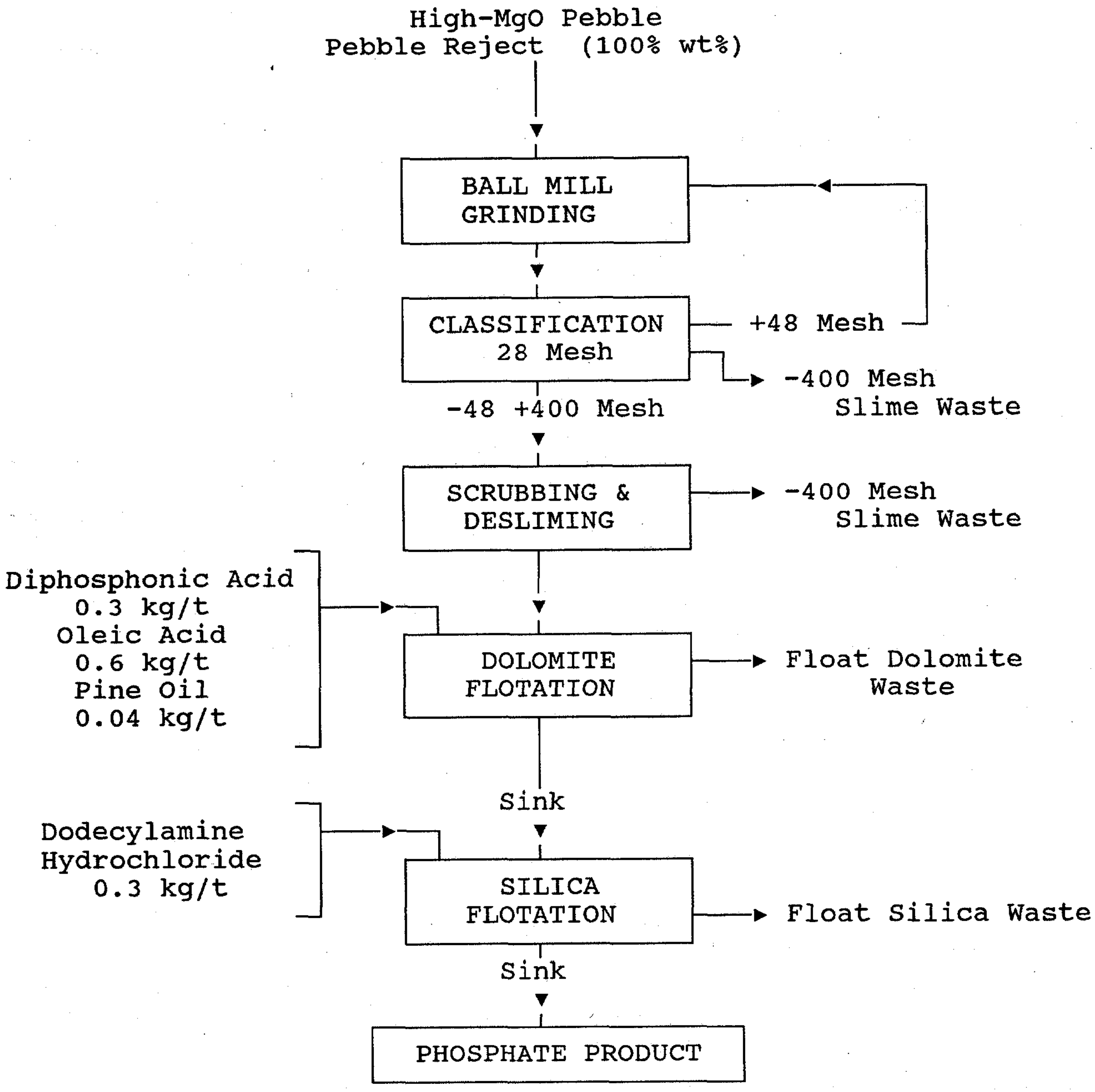

Table 15

T.V.A. Process Material Balance For 48/325 M Feed - Test 1

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	56.2	30.73	3.05	1.51	45.80	66.0	38.3
Silica Amine Tail.	10.3	1.76	93.24	0.11		0.7	0.4
Flot. -325 M Slime	0.6	25.29	7.62	3.09		0.6	0.9
Dolomite Tail.	0.6	21.39	13.44	4.44		0.5	1.3
-325 M Scrubber Slime	2.8	26.59	7.72	3.35		2.8	4.1
-325 M Rodmill Slime	29.5	26.15	5.84	4.14		29.4	55.0
Head Composite	100.0	26.18	13.38	2.22		100.0	100.0
Silica Flotation Feed	66.5	26.64	17.27	1.29		66.7	38.7
Dolomite Flotation Feed	67.7	26.19	16.90	1.34		67.8	40.9

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	83.1	30.73	3.05	1.51	45.80	97.5	93.7
Silica Amine Tail.	15.2	1.73	93.24	0.11		1.0	1.6
Flot. -325 M Slime	0.8	25.29	7.62	3.09		0.9	1.6
Dolomite Tail.	0.9	21.39	13.4	4.44		0.6	3.1
Flotation Feed	100.0	26.20	16.88	1.33		100.0	100.0

REAGENTS	Lb/T. Feed
Dequest 2010	0.71
Oleic Acid	1.32
Pine Oil	0.26
Azamine A36A	0.70
Diesel Fuel	0.30

Table 16

T.V.A. Process Material Balance For 48/325M Feed - Test 2

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	55.1	30.82	3.77	1.40	46.28	64.9	35.3
Silica Amine Tail.	10.4	2.34	91.63	0.12		0.9	0.5
Flot. -325 M Slime	0.4	24.78	8.00	2.54		0.4	0.5
Dolomite Tail.	1.3	20.13	15.65	4.62		1.0	2.7
-325 M Scrubber Slime	3.3	26.45	6.79	3.35		3.3	5.0
-325 M Rodmill Slime	29.5	26.15	5.84	4.14		29.5	56.0
Head Composite	100.0	26.16	13.78	2.18		100.0	100.0
Silica Flotation Feed	65.5	26.29	17.72	1.19		65.8	35.8
Dolomite Flotation Feed	67.2	25.63	17.62	1.27		67.2	39.0

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	82.0	30.82	3.77	1.40	46.28	96.6	90.1
Silica Amine Tail.	15.5	2.34	91.63	0.12		1.4	1.7
Flot. -325 M Slime	0.6	24.78	8.00	2.54		0.6	1.6
Dolomite Tail.	1.9	20.13	15.65	4.62		1.4	6.6
Flotation Feed	100.0	26.16	17.64	1.27		100.0	100.0

REAGENTS	Lb/T. Feed
Dequest 2010	0.71
Oleic Acid	2.02
Pine Oil	0.40
Azamine A36A	0.70
Diesel Fuel	0.30

Table 17

T.V.A. Process Material Balance For 48/400M Feed
Report of Previous Work

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	54.6	30.70	3.20	1.01		63.9	26.7
Silica Amine Tail.	11.6	5.00	83.60	0.27		2.2	1.5
Flot. -400 M Slime Dolomite Tail.	5.1	14.50	18.30	5.98		2.8	14.7
-400 M Scrubber Slime	7.3						
-400 M Rodmill Slime	21.4	28.50	4.70	4.10		31.1	57.1
Head Composite	100.0	26.30	13.70	2.06		100.0	100.0
Silica Flotation Feed	66.2	26.20	17.30	0.87		66.1	28.2
Dolomite Flotation Feed	71.3	25.40	17.40	1.23		68.9	42.9

	Analysis, %					% Distribution	
Product	% Wt.	P_2O_5	Insol	MgO	CaO	P_2O_5	MgO
Phosphate Conc.	76.6	30.70	3.20	1.01		92.7	62.6
Silica Amine Tail.	16.3	5.00	83.60	0.27		3.2	3.3
Flot. -400 M Slime Dolomite Tail.	7.1	14.50	18.30	5.98		4.1	34.1
Flotation Feed	100.0	25.40	17.40	1.23		100.0	100.0

REAGENTS	Lb/T. Feed
Dequest 2010	0.66
Oleic Acid	1.32
Pine Oil	0.09
Azamine A36A	0.66
Diesel Fuel	0.00

1.3. Flotation Reagent Consumption and Cost: Flotation reagent consumption and cost were calculated for all pertinent laboratory tests. Values were tabulated on a "per ton of initial flotation stage feed" basis. The total consumption and cost per ton of product was calculated by multiplying the "per ton of feed total values" by the flotation ratio of concentration. Results are presented in Tables 18-21. These tables also contain values calculated for the "optimized" results, presented in the reports prepared by original investigators, for comparison. A summary of the cost/ton of product and the product % MgO for each test is as follows:

Feed Size, Tyler Mesh	Process Used	Concentrate % MgO	Reagent Cost $/Ton Conc.
24/150	IMCF (previous)	0.86	2.86
28/150	IMCF T-1	0.81	2.68
	IMCF T-2	1.12	2.79
35/150	IMCF (Previous)	0.77	2.69
	IMCF T-3	0.84	2.60
	IMCF T-4	0.74	2.48
28/150	U.S.B.M. (Previous)	1.10	1.07
	U.S.B.M. T-2	1.22	2.37
	U.S.B.M. T-3	1.54	1.75
	U.S.B.M. T-4	1.66	2.01
35/150	UF (Previous)	1.10	(2.57)[9]
	UF T-2	0.97	4.02
	UF T-3	1.04	4.25
48/325	T.V.A. (Previous)	1.01	1.91
	T.V.A. T-1	1.51	2.00
	T.V.A. T-2	1.40	2.47

The reagent prices used to determine the cited processing costs were supplied by IMCF, local vendors, T.V.A., and American Cyanamid.

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[9] Approximation not including cost for unspecified quantity of Aerofroth 65 frother used at $0.865/lb.

Table 18
IMCF PROCESS REAGENT CONSUMPTION & COSTS

	24/150 Feed (Report)			35/150 Mesh Feed (Report)		
Reagent Name	Lb/T Feed	$/Lb	$/T Feed	Lb/T Feed	$/Lb	$/T Feed
AZ-36A	1.10	0.26	0.286	1.00	0.26	0.286
Diesel Fuel	0.60	0.10	0.060	0.50	0.10	0.050
Total			0.346			0.336
			1/Conc. Ratio (F) = 0.880			
Acetic Acid	0.79	0.20	0.158	0.76	0.20	0.152
Armac T	1.58	0.60	0.948	1.53	0.60	0.918
Diesel Fuel	4.75	0.10	0.475	4.58	0.10	0.458
Tergitol NP-10	0.12	0.74	0.189	0.10	0.74	0.074
Total			1.670			1.602
Grand Total			2.016			1.938
	Conc. Ratio = 1.42 $ 2.863/T PC			Conc. Ratio = 1.39 $ 2.694/T PC		

Table 18 (cont.)

Reagent Name	Test 1 Lb/T Feed	Test 1 $/Lb	Test 1 $/T Feed	Test 2 Lb/T Feed	Test 2 $/Lb	Test 2 $/T Feed
AZ-36A	1.2	0.26	0.312	1.2	0.26	0.312
Diesel Fuel	0.6	0.10	0.060	0.6	0.10	0.060
Total			0.372			0.372

1/Conc. Ratio (F) = 0.850

	Dolomite	Silica		Dolomite	Silica	Dolomite	Silica		Dolomite	Silica
Acetic Acid	(0.700)	0.603	0.20	(0.140)	0.121	(0.70)	0.603	0.20	(0.140)	0.121
Armac T	(1.600)	1.379	0.60	(0.960)	0.827	(2.00)	1.724	1.60	(1.200)	1.034
Diesel Fuel	(4.800)	4.138	0.10	(0.480)	0.414	(6.00)	5.172	0.10	(0.600)	0.517
Tergitol NP-10	(0.025)	(0.022)	0.74	(0.019)	0.016	(0.03)	0.026	0.74	(0.022)	0.019
Total				(1.599)	1.378				(1.962)	1.691
Grand Total					1.750					2.063

Conc. Ratio = 1/0.862 X 0.758 = 1.530
$2.678/T PC

Conc. Ratio = 1/0.862 X 0.857 = 1.354
$ 2.793/T PC

Table 18 (cont.)

	Test 3			Test 4		
Reagent Name	Lb/T Feed	$/Lb	$/T Feed	Lb/T Feed	$/Lb	$/T Feed
AZ-36A	1.4	0.26	0.364	1.4	0.26	0.364
Diesel Fuel	0.7	0.10	0.070	0.7	0.10	0.070
Total			0.434			0.434

1/Conc. Ratio (F) = 0.850

	Dolomite	Silica		Dolomite	Silica	Dolomite	Silica		Dolomite	Silica
Acetic Acid	(0.70)	0.595	0.20	(0.140)	0.119	(0.70)	0.595	0.20	(0.140)	0.119
Armac T	(1.80)	1.530	0.60	(0.080)	0.918	(1.60)	1.360	1.60	(0.960)	0.816
Diesel Fuel	(5.40)	4.590	0.10	(0.540)	0.459	(4.80)	4.080	0.10	(0.480)	0.408
Tergitol NP-10	(0.03)	(0.026)	0.74	(0.022)	0.019	(0.03)	0.026	0.74	(0.022)	0.019
Total				(1.782)	1.515				(1.602)	1.362
Grand Total					1.949					1.796

Conc. Ratio = 1/0.850 X 0.881 = 1.335
$2.602/T PC

Conc. Ratio = 1/0.850 X 0.853 = 1.379
$ 2.477/T PC

Table 19

U.S.B.M. PROCESS REAGENT CONSUMPTIONS & COSTS

REAGENT NAME	Lb/T Feed	$/Lb.	$/T. Feed
REPORT			
Sodium Hydroxide	1.0	0.12	0.120
Oleic Acid	1.0	0.42	0.420
IPC Fuel Oil	1.5	0.07	0.105
"N" Sodium Silicate	1.0	0.06	0.060
Total			0.705
	Conc. Ratio	= 1/.661	= 1.513
			$1.067 Ton PC
TEST 2			
Sodium Hydroxide	1.4	0.12	0.168
Oleic Acid	1.0	0.42	0.420
IPC Fuel Oil	1.5	0.07	0.105
"N" Sodium Silicate	1.0	0.06	0.060
Total			0.753
	Conc. Ratio	= 1/.317	= 3.154
			$2.375 Ton PC
TEST 3			
Sodium Hydroxide	1.50	0.12	0.180
Oleic Acid	1.50	0.42	0.630
IPC Fuel Oil	2.25	0.07	0.158
"N" Sodium Silicate	1.00	0.06	0.060
Total			1.028
	Conc. Ratio	= 1/.558	= 1.701
			$1.749 Ton PC
TEST 4			
Sodium Hydroxide	1.64	0.12	0.197
Oleic Acid	2.00	0.42	0.840
IPC Fuel Oil	3.00	0.07	0.210
"N" Sodium Silicate	1.00	0.06	0.060
Total			1.307
	Conc. Ratio	= 1/.650	= 1.538
			$2.010 Ton PC

Table 20

UF PROCESS REAGENT CONSUMPTIONS & COSTS

REAGENT NAME	Lb/T Feed	$/Lb.	$/T. Feed
REPORT			
Sodium Hydroxide	1.89	0.12	0.227
M-28B Tall Oil	5.50	0.20	1.100
IPC Fuel Oil	--	0.07	--
Sulfuric Acid	1.32	0.02	0.026
Aerofroth 65	N.A.	0.865	--
AZ-36A	--	0.26	--
"N" Sodium Silicate	--	0.06	--
Total			1.353

Estim. Conc. Ratio = 1.9
$2.57 Ton PC
(before silica flotation)

TEST 2			
Sodium Hydroxide	2.10	0.12	0.252
M-28B Tall Oil	5.50	0.20	1.100
IPC Fuel Oil	--	0.07	--
Sulfuric Acid	4.00	0.02	0.080
Aerofroth 65	--	0.865	--
AZ-36A	0.6	0.26	0.156
"N" Sodium Silicate	--	0.06	--.
Total			1.588

Conc. Ratio 1/.395 = 2.531
$4.019 Ton PC

TEST 3			
Sodium Hydroxide	3.10	0.12	0.252
M-28B Tall Oil	5.50	0.20	1.100
IPC Fuel Oil	1.10	0.07	0.077
Sulfuric Acid	5.80	0.02	0.116
Aerofroth 65	(0.30)	0.865	0.260
AZ-36A	0.50	0.26	0.130
"N" Sodium Silicate	(0.40)	0.06	0.024
Total			1.959

Conc. Ratio 1/.461 = 2.169
$4.249 Ton PC

Table 21

T.V.A. PROCESS REAGENT CONSUMPTIONS & COSTS

REAGENT NAME	**Lb/T Feed**	**$/Lb.**	**$/T. Feed**
REPORT			
Dequest 2010	0.660	1.08	0.713
Oleic Acid	1.320	0.42	0.554
Pine Oil	0.088	0.25	0.022
Dodecylamine *	0.660	(0.26)	0.172
Diesel Fuel	--	0.10	--.
Total			1.461

Conc. Ratio = .713/.546 = 1.306
$1.908 Ton PC

TEST 1			
Dequest 2010	0.710	1.08	0.767
Oleic Acid	1.320	0.42	0.554
Pine Oil	0.260	0.25	0.065
Dodecylamine *	0.700	0.26	0.182
Diesel Fuel	0.300	0.10	0.030
Total			1.598

Conc. Ratio = 1/.798 = 1.253
$2.002 Ton PC

TEST 2			
Dequest 2010	0.710	1.08	0.767
Oleic Acid	2.020	0.42	0.848
Pine Oil	0.400	0.25	0.100
Dodecylamine *	0.700	0.26	0.182
Diesel Fuel	0.300	0.10	0.030
Total			1.927

Conc. Ratio = 1/.781 = 1.280
$2.466 Ton PC

* Azamine A36A price used in place of the very expensive dodecylamine hydrochloride.

1.4. Power Consumption and Costs: The grindability of pebbles to different flotation feed sizes were determined according to Bond's grindability test procedure as described in Appendix B. These values and a cost of $.05/KWH were the basis of the grinding costs shown in Figure 6.

The scrubbing times and corresponding power consumption were calculated on the basis of currently power consumption values used by the industry during conditioning flotation feeds.

1.5. Concentrate Size Analyses: Dry screen analyses were performed on selected concentrates produced by the various processing techniques. Results are presented in Table 22. A comparison of these size analyses with the appropriate feed sample size analyses listed in Table 2 shows that both concentrate and feed have similar distributions except for the UF (T-3) products. This product has a finer size, distribution than the flotation feed due to less flotation of the +48 mesh particles during the first stage rougher flotation of phosphate using Westvaco M-28 B collector.

Figure 6

Comparison of power and reagent cost for different dolomite flotation processes.

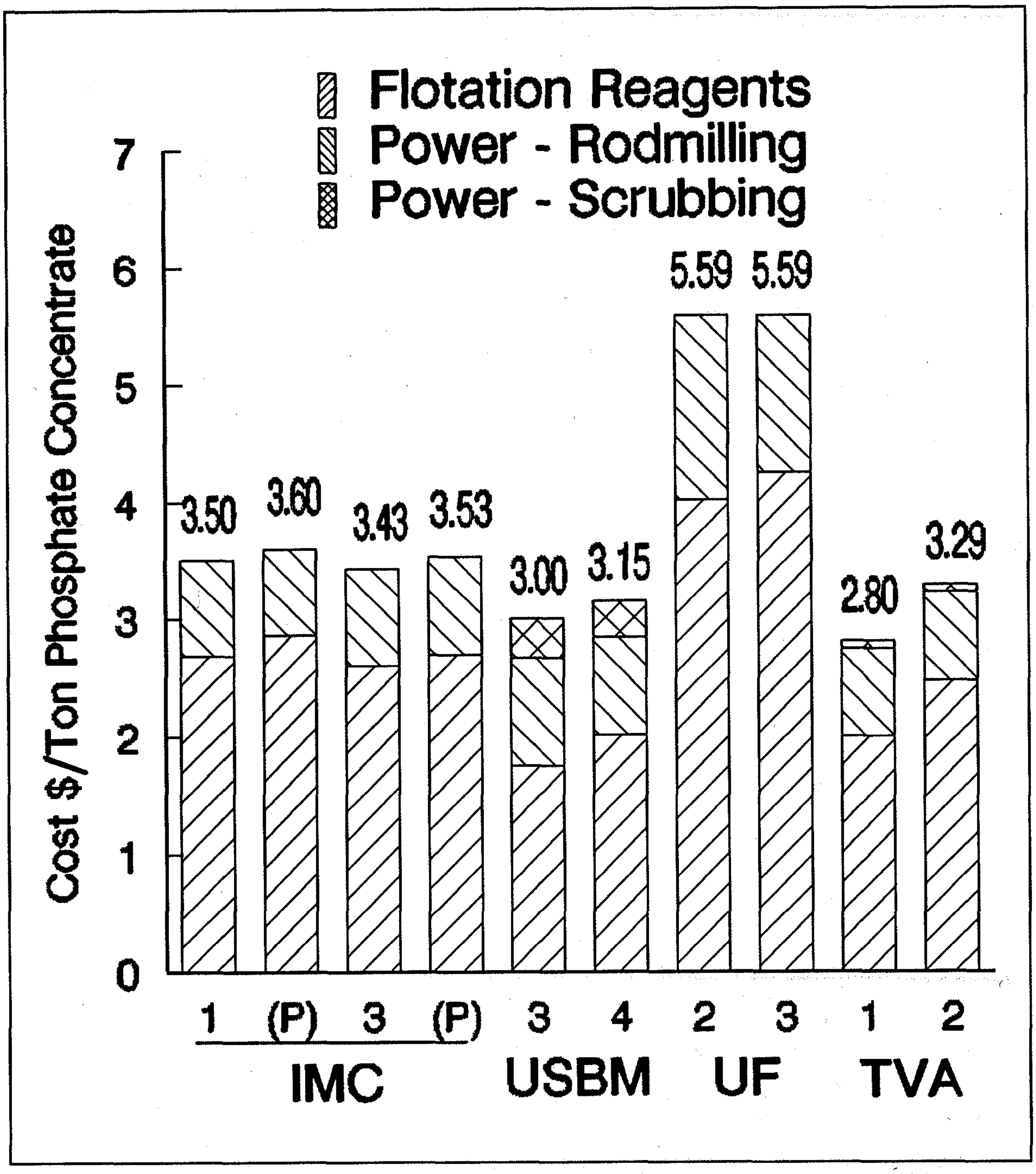


TABLE 22

SIZE ANALYSES OF SELECTED CONCENTRATES

TYLER MESH	% WT.	CUM. % WT.	% WT.	CUM. % WT.
	IMCF T-2 CONC.		USBM T-4 CONC.	
+28	0.1	0.1	Trace	Trace
28/35	31.2	31.3	29.7	29.7
35/48	25.2	56.5	26.0	55.7
48/65	19.6	76.1	20.7	76.4
65/100	12.2	88.3	12.8	89.2
100/150	9.3	97.6	9.6	98.8
-150	2.4	100.0	1.2	100.0
COMP.	100.0	--	100.0	--
	IMCF T-3 CONC.		Univ. of Fla. T-3 CONC.	
+35	0.9	0.9	Trace	Trace
35/48	30.2	31.1	19.1	19.1
48/65	31.6	62.7	36.4	55.5
65/100	18.8	81.5	23.0	78.5
100/150	14.7	96.2	16.9	95.4
-150	3.8	100.0	4.6	100.0
COMP.	100.0	--	100.0	--
	TVA T-2 CONC.			
+48	0.8	0.8		
48/65	34.5	35.3		
65/100	23.6	58.9		
100/150	19.1	78.0		
150/200	9.7	87.7		
200/325	10.9	98.6		
-325	1.4	100.0		
COMP.	100.0	--		

1.6. <u>Performance and Cost Comparisons:</u> The total and differential costs/ton of concentrates produced by different processes are shown in Figure 6. The performance indicators (flotation recovery and % Mgo in the concentrates) are given in Figure 7. It can be seen that IMC process produces concentrates of MgO values lower than 1.0% and lower than that of other processes. The cost per ton of concentrate in IMC process is somewhat higher than that of some of the other processes. However, since the products of these processes are not of acceptable values the costs should not be used as basis of comparison.

The only other process which produced concentrates of low MgO values is that of UF, but at much lower recoveries which is reflected in the high cost per ton of concentrate.

2-Hydrocyclone Testing

2.1. <u>Procedure and Results:</u> A total of nine 55 gallon drums were received by Met Pro containing ground, high MgO content phosphate rock. These samples were mixed in units of three with one drum from each unit then mixed and split into three equal sized samples. The composite sample analyzed 22.49% P_2O_5, 37.22% CaO, and 3.27% MgO. The screen analysis indicated that 35.8% was -400 mesh (37 microns) which, at a MgO content of 6.16%, contained 67.4% of the total MgO available. The screen fractions from 28 mesh down to +400 mesh were relatively consistent in MgO content (1.4-1.9% MgO) as shown below.

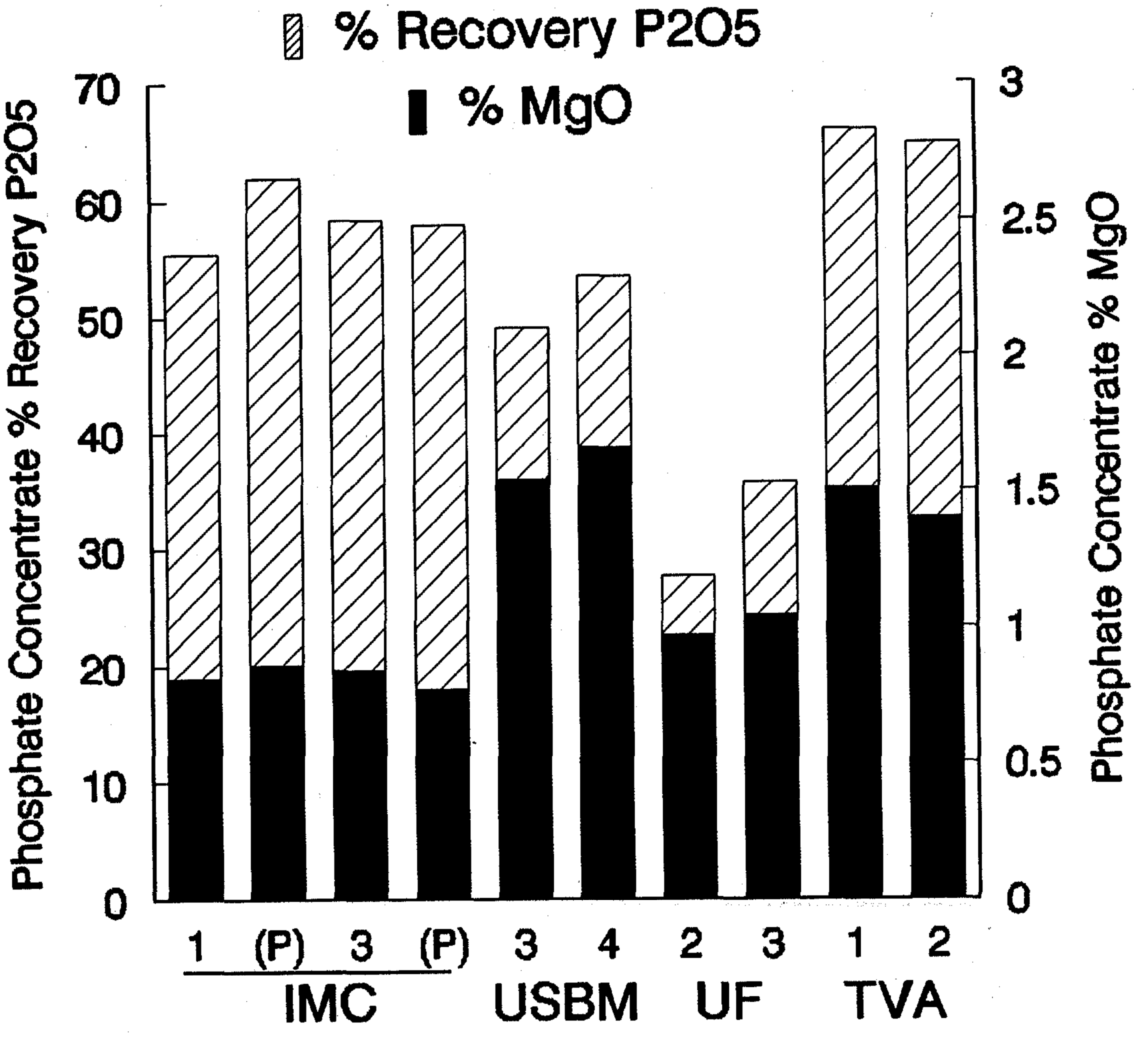
Feed Samples

Size,mesh	Weight%	%P205	%MgO	%CaO	%Insol
+ 28	0.3	23.68	1.71	37.27	18.83
-28+35	5.9	24.72	1.89	38.73	16.14
-35+48	12.0	24.00	1.93	37.70	17.58
-48+65	13.4	22.12	1.74	34.48	22.68
-65+100	13.0	22.11	1.55	33.86	25.20
-100+150	9.8	24.02	1.42	36.25	22.09
-150+200	4.8	25.61	1.40	38.29	18.19
-200+270	3.5	26.46	1.47	39.05	15.15
-270+325	1.4	26.32	1.90	39.91	12.75
-325+400	0.1	23.82	1.57	36.65	NS
-400	35.8	20.52	6.16	38.90	5.16

A series of cyclone tests, using the Met Pro six inch pilot circuit, was conducted. The cyclone was operated in a closed loop with the cyclone products recycled back into the same tank. Samples were taken periodically and sizing characteristics observed.

Figure 7

Comparison of concentrate grade obtained by different flotation processes



Underflow and overflow volume rates were measured under a variety of conditions. Samples were taken for screen analysis and percent solids determinations. To properly evaluate the MgO rejection by use of the cyclone a mass flow balance had to be developed for each operating condition. Samples taken for chemical analysis were wet screened at 400 mesh with the plus 400 mesh fraction dried and rescreened. All fractions were sent for chemical analyses as shown below.

Overflow Samples

Size,mesh	Weight%	%P_2O_5	%MgO	%CaO	%Insol
+ 28	TR	NA	NA	NA	
-28+35	0.1	20.38	1.22	32.92	23.96
-35+48	0.2	10.34	0.80	17.54	11.70
-48+65	0.3	20.31	1.61	33.58	26.80
-65+100	0.4	20.77	1.57	33.99	29.29
-100+150	0.4	21.75	1.49	35.75	26.11
-150+200	0.5	23.59	1.34	37.51	22.55
-200+270	0.7	25.51	1.22	39.76	18.50
-270+325	0.3	26.54	1.28	40.97	15.37
-325+400	0.1	24.78	1.24	39.07	-
-400	97.0	21.48	6.18	41.89	5.19

Underflow Samples

Size,mesh	Weight%	%P_2O_5	%MgO	%CaO	%Insol
+ 28	3.0	22.49	0.86	34.37	27.42
-28+35	28.1	25.14	1.55	39.46	18.88
-35+48	27.9	23.52	1.66	38.35	20.39
-48+65	17.4	22.65	1.69	36.40	24.26
-65+100	10.2	21.99	1.57	35.45	26.61
-100+150	5.0	22.61	1.42	35.80	25.96
-150+200	2.7	24.56	1.31	38.54	20.35
-200+270	1.6	25.50	1.34	39.59	17.86
-270+325	0.1	-	-	-	-
-325+400	TR	-	-	-	-
-400	4.0	22.32	5.31	41.85	7.27

The best test results obtained were with a 9.2% solids slurry feed, 1.625 inch vortex finder, 0.75 inch apex while operating at a pressure of about 15 PSI. The weight distribution and screen analysis indicated that 99.7% of the available +400 mesh fraction reported to the underflow. Of the -400 mesh present 88.2% reported to the overflow. The calculated head sample had 3.00% MgO vs. 3.27% in the composite feed. This specific test sample contained 64.2% of the total MgO in the -400 mesh fraction of the feed. The cyclone overflow contained 57.0% of this available MgO. The chemical analyses of the -400 mesh fractions of overflow and underflow, in all tests, showed that the overflow consistently contained a higher MgO content than the underflow, (i.e., 5.95 vs. 4.87%) as given below.

Overflow Samples

Size,Mesh	%P_2O_5	%MgO	%CaO	%insol
+400	22.57	1.33	36.12	22.01
-400	20.29	5.95	40.01	4.51

Underflow samples

Size,Mesh	%P_2O_5	%MgO	%CaO	%insol
+400	22.94	1.57	37.28	22.48
-400	22.23	4.87	40.14	8.85

The optimized cyclone test indicated that 28.6% of the total weight reporting to the overflow, which contained the following distribution of specific components.

Component	P_2O_5	CaO	MgO	LOI	CO2	Insol	Fe_2O_3	Al_2O_3
% Rejected	27.6	29.9	57.0	46.8	48.2	18.1	74.3	36.8

1.3. Concluding Remarks: The size distribution of all samples analyzed did not indicate any significant difference in the MgO content from the most coarse size of +28 mesh down to +325 mesh. Grinding did not significantly effect the ratio of P_2O_5 to MgO until finer than 400 mesh. The P_2O_5 content of the -400 mesh did not vary from the coarser sizes.

It is apparent that there are two distinct forms of MgO in these particular samples. The MgO as dolosilt seems to breakup into its natural silt size very easily when ground. This is the material that appears to report to the -400 mesh fraction.

The cyclone appears to be an excellent tool to remove dolosilt after it has been ground. The harder dense dolomite does not appear to be preferentially ground so no apparent gain is made by separation by sizing methods. The distribution of these two components can only be interpreted but if we assume that the hard dolomite-phosphate ratio stays consistent within the -400 mesh fraction then approximately 47% of the total MgO content of this rock is in the form of dolosilt.

The fines resulting from grinding and cyclone separation contain about 6.0% MgO and over 20.0% P_2O_5. This material, starting at an initial 3.0% solids, consolidated to about 36% solids in about two weeks. Because of its apparent high specific surface area it would appear that the P_2O_5 content would be relatively available if utilized as a direct application rock.

3- Selective Flocculation Tests

Tests were conducted according to the procedure described in the progress report of Ohio State University project(FIPR # 89-02-083). Six grams of -325 mesh phosphate pebble material was used in 200 ml of solution to give 3.0% by weight. The following experimental conditions were used:

- Dispersant: Sodium silicate at 100 ppm.
- Flocculant : PAM-Tiron Mix at 1.0 and 0.5 ppm.
- 500 ml beaker and 2.0 in. dia. propeller.
- Dispersion at 350 rpm for 2.0 minutes in sodium silicate solution (pH = 8.8).
- Flocculant added at 350 rpm for 2.0 minutes.
- Conditioning at 150 rpm for 4.0 minutes.
- Stop agitation for 2.0 minutes and syphon supernatant.
- Dry settled material (flocculated) and send for chemical analyses.

Two tests were conducted using 1.0 ppm of polymer and the results indicated complete flocculation of the material used indicating bulk flocculation rather than selective flocculation.

Two more tests were carried out using 0.5 ppm of the polymer. Only 90% of the feed material was flocculated in these tests. Nevertheless, the analyses of the settled(flocculated) material indicated similar analyses to the feed ore as shown below.

Chemical Analyses of flocculated Material

Test#	% P_2O_5	%MgO	% CaO	% Insol.
1	26.5	2.38	49.08	10.58
2	26.2	2.37	49.74	10.25

Again the results suggest bulk flocculation. The unselectivity of flocculation was also noticed by Ohio state investigators and was attributed to slime coating and poor dispersion. This may suggest that further work is needed in this area before selective flocculation is recommended as a processing technique for dolomitic phosphate ores.

GENERAL CONCLUSIONS

* It is apparent that there are two distinct forms of MgO in these particular samples. The MgO as dolosilt seems to breakup into its natural silt size very easily when ground. This is the material that appears to report to the -400 mesh fraction.

* The reduction of MgO as dolosilt by grinding rock and desliming with a cyclone appears to be a cost effective method.

* The cyclone overflow material resulting from grinding should not be considered as waste, but one that has some potential as an alternate fertilizer product. It has a relatively high P_2O_5 content, high surface area for quick release and contains sufficient fine grained MqO that could be applicable for a variety of uses.

* To further reduce the MgO content by removing the hard dolomite will require other concentration methods such as flotation.

* Five flotation processes were tested by their developers.The reported results indicate that most of these techniques produced concentrates of MgO content about 1.0% or less.

* The above processes were tested at FIPR's laboratory (under their reported optimum conditions) using the same raw material from phosphate pebbles. The test results, however, did not duplicate most of the results obtained by the developers of these processes. This has been attributed, in some cases, to the difference in water hardness used by different researchers. This has been very much evident with the anionic processes (using fatty acids as collectors).

* Performance and cost comparisons indicate that IMCF process produces concentrates of MgO values lower than the other processes and at high recovery values. Even though the operating cost is higher than others,it can be considered the most cost effective process due to the lack of need for grinding and/or scrubbing equipment as in some of the other processes. In addition, most of the anionic processes require close pH control and probably water softening systems, which makes their adaptation more difficult and costly.

* Pilot plant testing of IMCF cationic process is strongly recommended.

* Preliminary selective flocculation tests did not produce any measurable separation of dolomite from phosphate and this was attributed to poor dispersion of slimes. More work is recommended if this process is to be used with very fine ores.

APPENDIX A

REPORTED RESULTS OF DIFFERENT INVESTIGATORS

1-IMCF
2-UF
3-UA
4-U.S.B.M.
5-TVA

IMC FERTILIZER, INC.

FLORIDA INSTITUTE
OF
PHOSPHATE RESEARCH
1993 JAN 19 PM 12:06

January 19, 1993

Dr. Hassan El-Shall
Florida Institute of
Phosphate Research
1855 W. Main Street
Bartow, FL 33830

Dear Hassan:

The planned testwork for this project has been completed by IMCF. Referring to your August 24, 1992 memo containing six general instructions to the various investigators, the following results and comments are submitted for comparison/evaluation by FIPR:

(1) The IMC process used to process the 2+% MgO pebble sample is described in detail in the following:

U. S. Patent 4,144,969; Snow, R. E.; Beneficiation of Phosphate Ore; March 20, 1979.

U. S. Patent 4,189,103; Lawver, J. E.; Snow, R. E. and McClintock, W.O.; Method of Beneficiating Phosphate Ores; February 19, 1980.

Two flotation separation tests were performed using 24/150 mesh feed, and one follow-up test was performed using 35/150 mesh feed prepared by batch rodmilling and desliming by pulping/decantation over a 150 mesh screen.

(2) The total processing flowsheet, in simplified form, is shown in Figure 1. Overall material balances for the best tests are presented in Table 1. Balances for the silica flotations and for the phosphate-dolomite separations alone are presented in Table 2.

(3) Reagent names and usage rates are listed in Table 3. Silica (amine) flotation was performed using standard lab procedure.

Conditioning was performed for 20 seconds at about 70% solids for the phosphate-dolomite separation stage. Final conditioning pH was 5.2-5.4 for the various tests performed. Flotation time per stage was 1.5 minutes or less using 500 g. feed charges. Tallow amine use was 1.80 bl./ton feed for tests 2 and 10, and 1.52 lb./ton feed for test 1. Diesel:amine ratio was 3:1 for all tests.

(4) Size/assay analyses for feed and phosphate concentrate are presented in Table 4 for -24 mesh and -35 mesh grinds.

IMC Fertilizer, Inc., Minerals Operations, P.O. Box 867, Bartow, Florida 33830, (813) 533-1121

(5) Tallow amine, acetic acid*, and diesel are present in conventional flotation plant waters as a result of their addition to the amine circuit. These reagents are not considered as environmentally damaging using current water recycling systems. Tergitol NP-10 is a nonionic ethoxylated nonylphenol used as a frothing agent. An MSDS is attached for reference to toxicity.

(6) Testing of the IMCF cationic process for phosphate-dolomite separation has been performed in the lab on more than fifty different pebble and/or concentrate samples since the mid-1970's. The process works using feed as coarse as 24 mesh. Phosphate concentrates assaying >66% BPL and <1.1% MgO were consistently produced from phosphate/dolomite separation feeds analyzing as high as about 3.2% MgO. Brief pilot plant tests conducted by John Keating at our Noralyn lab confirmed the technical feasibility of the process using flotation columns. In continuous pilot plant operation, the dolomite recleaner and cleaner tailings should be sized at about 48 mesh, and the +48 mesh fraction is recycled to the dolomite separation feed conditioner to obtain the highest possible BPL recovery. The -48 mesh fraction (high % MgO) is discarded.

Total flotation reagent cost per ton of phosphate concentrate produced from the test sample is estimated to be $3.00±0.11, of which about $.0.50 was required for silica pre-flotation.

Sincerely,

R.E. Snow

R. E. Snow

mc
attachments

*As acetate ion from the neutralized amine used.

FIGURE 1
SIMPLIFIED PEBBLE PROCESSING FLOWSHEET
IMCF CATIONIC PROCESS

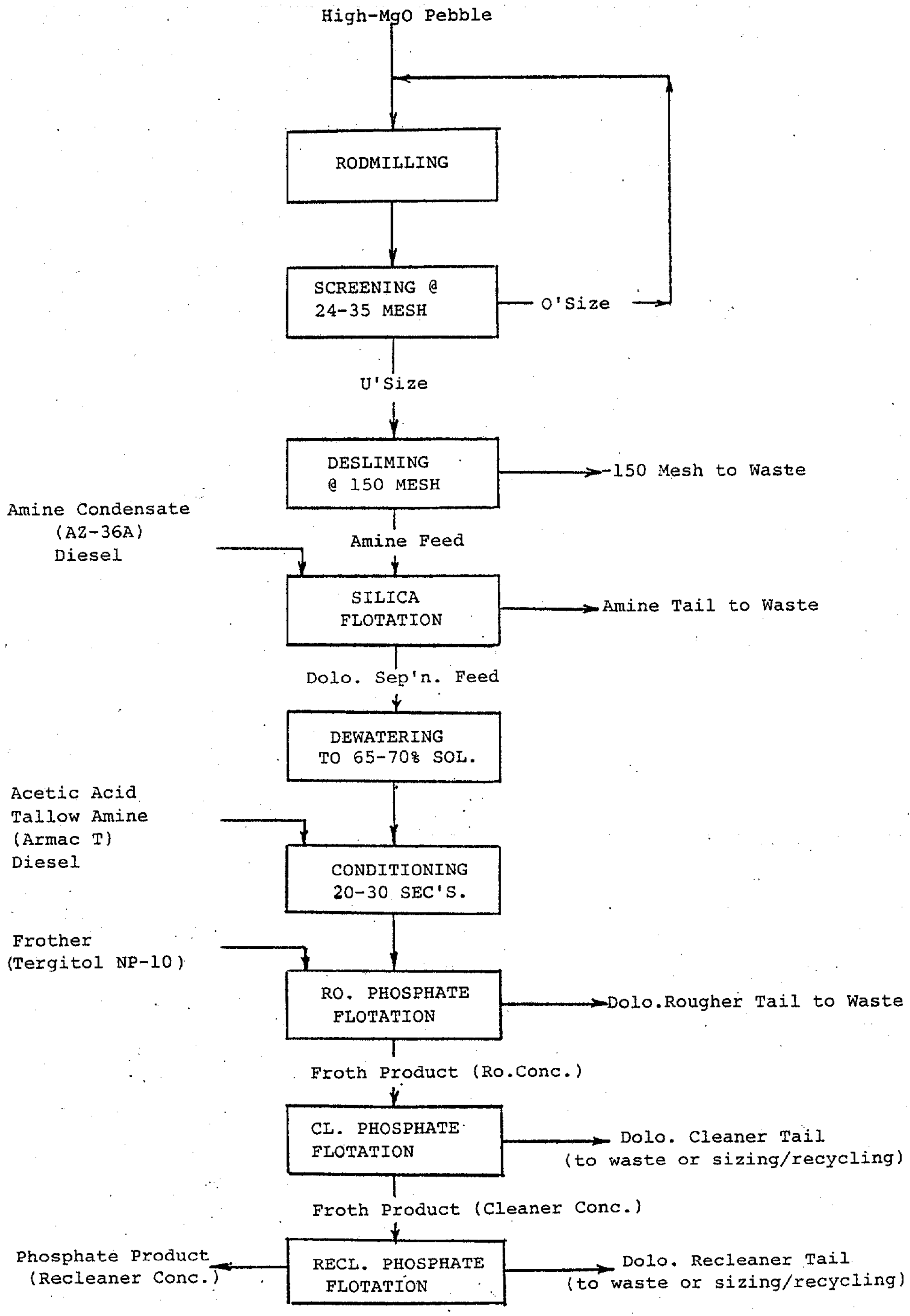


TABLE 1

OVERALL PEBBLE PROCESSING MATERIAL BALANCES

		ANALYSIS, %			% DISTRIBUTION		
PRODUCT	% WT.	BPL	INSOL	MgO	BPL	INSOL	MgO
24 M Grind:							
Phos. Conc.	52.1	67.80	5.45	0.86	61.9	21.6	21.8
Dolo. CL/RCL. Tail	7.7	58.64	3.98	3.47	7.9	2.4	13.1
Dolo. Ro. Tail	5.5	44.12	4.56	7.40	4.2	1.9	19.9
Silica Am. Tail	8.9	5.46	90.62	0.16	0.9	61.3	0.5
-150M R.M. Slime	25.8	55.63	6.55	3.56	25.1	12.8	44.7
Composite	100.0	(57.10)	(13.16)	(2.06)	100.0	100.0	100.0
Amine Feed	74.2	(57.61)	(15.46)	(1.54)	74.9	87.2	55.3
Dolomite Sep'n Feed	65.3	(64.72)	(5.21)	(1.73)	74.0	25.9	54.8
35 M Grind:							
Phos. Conc.	47.5	69.11	3.02	0.77	57.9	11.1	18.6
Dolo. CL/RCL Tail	5.1	57.25	2.64	3.74	5.2	1.0	9.5
Dolo.Ro. Tail	3.4	34.38	3.52	9.55	2.1	1.0	16.6
Silica Am. Tail	10.0	5.93	88.97	0.21	1.0	69.0	1.0
-150 R.M. Slime	34.0	56.34	6.79	3.18	33.8	17.9	54.3
Composite	100.0	(56.67)	(12.89)	(1.99)	100.0	100.0	100.0
Amine Feed	66.0	(56.83)	(16.03)	(1.38)	66.2	82.1	45.7
Dolomite Sep'n Feed	56.0	(65.93)	(3.00)	(1.59)	65.2	13.1	44.7

TABLE 2

SILICA FLOTATION MATERIAL BALANCES

		ANALYSIS, %			% DISTR.	
PRODUCT	% WT.	BPL	INSOL	MgO	BPL	MgO
24M GRIND:						
Amine Conc.	88.0	64.34	5.27	1.72	98.8	98.7
Amine Tail	12.0	5.46	90.62	0.16	1.2	1.3
Amine Feed	100.0	(57.28)	(15.51)	(1.53)	100.0	100.0
35 M GRIND:						
Amine Conc.	84.8	65.92	3.01	1.57	98.4	97.8
Amine Tail	15.2	5.93	88.97	0.21	1.6	2.2
Amine Feed	100.0	(56.80)	(16.07)	(1.36)	100.0	100.0

PHOSPHATE/DOLOMITE SEPARATION MATERIAL BALANCES

			ANALYSIS, %			% DISTR.	
TEST	PRODUCT	% WT.	BPL	INSOL	MgO	BPL	MgO
	24M Grind:						
2	Phos. Conc.	79.8	67.80	5.45	0.86	83.6	40.1
	Dolo. Recl. Tail	6.5	62.36	3.90	2.58	6.2	9.9
	Dolo. Cl. Tail	5.3	54.15	4.12	4.54	4.4	14.0
	Dolo. Ro. Tail	8.4	44.12	4.56	7.40	5.8	36.0
	Composite	100.0	(64.73)	(5.20)	(1.72)	100.0	100.0
	Cleaner Conc.	86.3	(67.38)	(5.33)	(0.99)	89.8	50.0
1	Phos. Conc.	69.5	67.60	5.85	0.76	73.2	30.6
	Dolo. Recl. Tail	8.9	65.48	3.74	1.51	9.1	7.5
	Dolo. Cl. Tail	8.0	59.87	3.84	3.09	7.4	14.5
	Dolo. Ro. Tail	13.6	48.53	4.37	6.00	10.3	47.4
	Composite	100.0	(64.20)	(5.29)	(1.73)	100.0	100.0
	Cleaner Conc.	78.4	(67.36)	(5.60)	(0.84)	82.3	38.1
	35 M Grind:						
10	Phos. Conc.	84.8	69.11	3.02	0.77	88.9	41.4
	Dolo. Recl. Tail	5.2	62.04	2.48	2.56	4.9	8.3
	Dolo. Cl. Tail	3.9	50.89	2.89	5.35	3.0	13.4
	Dolo. Ro. Tail	6.1	34.38	3.52	9.55	3.2	36.9
	Composite	100.0	(65.92)	(3.01)	(1.57)	100.0	100.0
	Cleaner Conc.	90.0	(68.71)	(2.99)	(0.87)	93.8	49.7

TABLE 3

LAB FLOTATION REAGENT USAGE

Feed Tyler Mesh	Reagent	Lb./T Silica Flot. Feed	Lb./T Dolo.Sep'n. Feed	Lb./T. Phos. Conc.
	Silica Flot:			
24/150	AZ-36A	1.10	1.25	1.56
24/150	Diesel	0.60	0.68	0.85
35/150	AZ-36A	1.00	1.18	1.39
35/150	Diesel	0.50	0.59	0.70
	Dolo. Sep'n.:			
24/150	Acetic Acid	0.79	0.90	1.12
24/150	Armac T	1.58	1.80	2.24
24/150	Diesel	4.75	5.40	6.75
24/150	Tergitol NP-10	0.12	0.14	0.17
35/150	Acetic Acid	0.76	0.90	1.06
35/150	Armac T	1.53	1.80	2.13
35/150	Diesel	4.58	5.40	6.37
35/150	Tergitol NP-10	0.10	0.14	0.14
24/150	Total Reagents	8.94	10.17	12.70
35/150	Total Reagents	8.47	10.01	11.77

24/150 Conc. Ratio - Phos.Conc./Silica Flot. Feed = 1.42 F/C
35/150 Conc. Ratio - Phos.Conc./Silica Flot. Feed = 1.39 F/C

TABLE 4

FLOTATION FEED SIZE/ASSAY ANALYSIS

PRODUCT	TYLER MESH	% WT.	ANALYSIS, %		
			BPL	INSOL	MgO
	24 M Grind:				
Amine Feed	+24	0.8	57.98	14.13	1.33
	24/28	11.9			
	28/35	30.0	59.33	12.29	1.55
	35/48	19.9	57.96	13.36	1.70
	48/65	16.0	53.25	18.82	1.63
	65/100	9.4	52.14	21.02	1.51
	100/150	8.0	53.30	19.30	1.52
	-150	4.0	57.82	13.04	1.71
	Composite	100.0	(57.15)	(15.31)	(1.58)
	Head Cut		56.57	15.71	1.56
	35 M Grind:				
Amine Feed	+35	0.2	57.65	14.15	1.32
	35/48	31.4			
	48/65	31.9	56.53	15.50	1.46
	65/100	17.0	55.25	18.05	1.46
	100/150	13.4	55.74	17.69	1.39
	-150	6.1	58.12	13.81	1.53
	Composite	100.0	(56.77)	(15.72)	(1.42)
	Head Cut		56.89	16.29	1.43

FLOTATION CONCENTRATE SIZE/ASSAY ANALYSIS

PRODUCT	TYLER MESH	% WT.	ANALYSIS,%			% DISTRIBUTION	
			BPL	INSOL	MgO	BPL	MgO
	24 M Grind:						
2 Phos. Conc.	+28	10.4	64.30	10.36	0.54	10.0	6.8
	28/35	28.6	67.36	6.56	0.63	28.8	21.9
	35/48	19.1	66.56	5.49	0.74	19.0	17.2
	48/65	17.2	67.26	4.50	0.92	17.3	19.2
	65/100	10.1	67.58	3.27	1.06	10.2	13.0
	100/150	9.4	67.48	2.92	1.17	9.5	13.4
	-150	5.2	66.61	2.60	1.34	5.2	8.5
	Composite	100.0	(66.86)	(5.51)	(0.82)	100.0	100.0
	Head Cut		67.80	5.45	0.86		
	35 M Grind:						
10 Phos. Conc.	+48	26.8	68.62	4.20	0.66	26.7	22.9
	48/65	31.7	68.71	3.38	0.75	31.6	30.8
	65/100	17.6	69.43	2.50	0.82	17.7	18.7
	100/150	15.2	69.28	2.11	0.85	15.3	16.7
	-150	8.7	68.53	2.34	0.97	8.7	10.9
	Composite	100.0	(68.88)	(3.16)	(0.77)	100.0	100.0
	Head Cut		69.11	3.02	0.77		

UNION CARBIDE

MATERIAL SAFETY DATA SHEET

EFFECTIVE DATE: APRIL 30, 1980

I. IDENTIFICATION

PRODUCT NAME:	TERGITOL® NONIONIC SURFACTANT NP-10		
CHEMICAL NAME:	Nonylphenol Polyethylene Glycol Ether	CHEMICAL FAMILY:	Nonionic Surfactant
FORMULA:	$C_{36}H_{66}O_{10}$	MOLECULAR WEIGHT:	682 (average)
SYNONYMS:	An alkylphenyl-hydroxypolyoxyethylene		
DEPARTMENT OF TRANSPORTATION	HAZARD CLASSIFICATION	None	
	SHIPPING NAME	None	
CAS # 26027-38-3	CAS NAME	Poly(oxy-1,2-ethanediyl), *alpha*-(4-nonylphenol)-*omega*-hydroxy-	

II. PHYSICAL DATA

BOILING POINT, 760 mm. Hg	>250°C. (>482°F.) with decomposition	FREEZING POINT	7°C.
SPECIFIC GRAVITY (H_2O = 1)	1.062 at 20/20°C.	VAPOR PRESSURE at 20°C.	<1 mm. Hg
VAPOR DENSITY (air = 1)	>1	SOLUBILITY IN WATER, % by wt.	Miscible
PER CENT VOLATILES BY VOLUME	Nil	EVAPORATION RATE (Butyl Acetate = 1)	<0.01
APPEARANCE AND ODOR	Clear, slightly yellow liquid; mild and characteristic odor.		

III. INGREDIENTS

MATERIAL	%	TLV (Units)	HAZARD
Nonylphenol ethoxylate	100	Not established	Irritant

IV. FIRE AND EXPLOSION HAZARD DATA

FLASH POINT [test method(s)]	500°F., Cleveland open cup ASTM D 92
FLAMMABLE LIMITS IN AIR, % by volume	Not determined (Nonvolatile material)
EXTINGUISHING MEDIA	Use water spray, carbon dioxide, dry chemical, alcohol-type or universal-type foams applied by manufacturers' recommended technique.
SPECIAL FIRE FIGHTING PROCEDURES	None
UNUSUAL FIRE AND EXPLOSION HAZARDS	None

EMERGENCY PHONE NUMBER

304/744-3487

This number is available days, nights, weekends, and holidays.

UNION CARBIDE CORPORATION • ETHYLENE OXIDE DERIVATIVES DIVISION
OLD RIDGEBURY ROAD, DANBURY, CT 06817

V. HEALTH HAZARD DATA

TLV AND SOURCE: None established by ACGIH or OSHA

ACUTE EFFECTS OF OVEREXPOSURE	
SWALLOWING	May cause nausea, vomiting, and diarrhea.
SKIN ABSORPTION	None currently known.
INHALATION	None currently known. Mists may cause chest discomfort and coughing.
SKIN CONTACT	Prolonged contact as from clothing wet with the chemical may cause irritation. Contact with bare skin — no problem.
EYE CONTACT	Severe irritation.
CHRONIC EFFECTS OF OVEREXPOSURE	None currently known.
OTHER HEALTH HAZARDS	None currently known.

EMERGENCY AND FIRST AID PROCEDURES:	
SWALLOWING	Give 2 glasses of water and induce vomiting by putting finger down throat. Call a physician.
SKIN	Remove contaminated clothing and flush with water.
INHALATION	If symptoms should develop, remove to fresh air. Call a physician.
EYES	Immediately flush eyes with plenty of water for at least 15 minutes. Call a physician.

NOTES TO PHYSICIAN

This product will act like any nonionic surfactant. It may be advisable to seek ophthalmologic consultation for eye injury.

VI. REACTIVITY DATA

STABILITY			
UNSTABLE	STABLE	CONDITIONS TO AVOID	None
—	√		

INCOMPATIBILITY (materials to avoid)	None
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS	Burning can produce carbon dioxide or carbon monoxide.

HAZARDOUS POLYMERIZATION			
May Occur	Will not Occur	CONDITIONS TO AVOID	None
—	√		

VII. SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED	Wear eye protection. Collect for disposal. Highly toxic to fish. Avoid discharge to natural waters.
WASTE DISPOSAL METHOD	Incinerate in a furnace where permitted under appropriate Federal, State, and local regulations.

VIII. SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION (specify type)	None required		
VENTILATION	Normal room ventilation is considered satisfactory.		
PROTECTIVE GLOVES	Plastic	EYE PROTECTION	Monogoggles
OTHER PROTECTIVE EQUIPMENT	Eye bath and safety shower		

IX. SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING

Avoid contact with eyes.
Wash thoroughly after handling.

FOR INDUSTRY USE ONLY

OTHER PRECAUTIONS

This product is not readily biodegradable in a wastewater treatment system and is highly toxic to aquatic life. The preferred method of disposal is incineration.

Progress Report

Bench-Scale Optimization of the Two Stage Conditioning Process for Removal of Dolomite from Apatite

(FIPR Grant # 89-02-082S)

Submitted to:

Florida Institute of Phosphate Research
1855 W. Main Street
Bartow, Fl 33830

Submitted by:

Brij M. Moudgil
Mineral Resources Research Center
Department of Materials Science and Engineering
University of Florida
Gainesville, Florida 32611

August 1993

SUMMARY

Separation of dolomite from the high dolomitic pebble feed supplied by FIPR was investigated using the two stage conditioning process. Optimal separation was achieved by conditioning the feed with 2.5 kg/t fatty acid collector at pH 10, reconditioning at pH 4 followed by dolomite flotation at pH 5. Under these conditions MgO content decreased by 43% from 1.27% MgO in the deslimed feed. On an insol-free basis the MgO content of the concentrate is 0.83% which is below the desired limit of 1% MgO. The P_2O_5 content of the concentrate analyzed 25.14% (54.9% BPL) at about 90% recovery level. No attempt was made in this study to separate sillica from apatite. On an insol-free basis the P_2O_5 content would be 29% (63.3% BPL). Considering the high grade of the phosphate rock feed the fatty acid consumption may be economical. The reagent amount for the 35x150 feed needs to be optimized to improve the P_2O_5 recovery.

INTRODUCTION

A selective dolomite flotation process involving a two-stage conditioning technique was previously developed at the University of Florida. These investigations initially involved studies on synthetic apatite-dolomite mixtures in a microflotation (Hallimond) cell [1]. The collectors employed were of laboratory reagent grade and flotation was carried out in a deionized water medium. The suitability of this technique under industrial conditions of operation was demonstrated by later studies conducted on the bench scale using naturally occurring high dolomitic phosphate rock samples, commercially available reagents, and industrial quality water [2].

The aim of the present work was to establish the feasibility of the two-stage conditioning technique using a given dolomitic phosphate rock sample in a round robin study conducted by the Florida Institute of Phosphate Research (FIPR).

EXPERIMENTAL

Materials

Flotation feed: The as-received 5x20 mesh pebble feed from FIPR was pulverized and screened to collect different size fractions. In all the tests 65x150 mesh fraction was used unless mentioned otherwise. The feed was deslimed by repeated high speed mixing and decantation. About 9% material was lost during the desliming process. Chemical analysis of the different size fractions and the deslimed feed is given in Table 1. It is seen that the 65x150 mesh fraction has a lower MgO content than both the pebble and the 35x65 fraction. Further, the MgO content decreased in the deslimed flotation feed. The lowering of MgO content in the flotation feed is attributed to the lower hardness of dolomite compared with silica and apatite which results in greater degree of attritioning of dolomite during the pulverization and desliming steps.

It was shown in an earlier study [2] that the two stage conditioning process is ineffective in separating palygorskite which in some phosphate ores is a major source of magnesium. X-ray diffraction of the slime fraction revealed that the ore consisted of apatite, quartz and dolomite. Diffuse reflectance FT-IR also did not reveal the presence of any clay-like material in the feed.

Reagents: Commercially available reagents were used during bench-scale testing. Fatty acid (M-28B) and fuel oil were obtained from Westvaco Chemical Co. AeroFroth 65 (AF-65) frother was procured from American Cyanamid Co. The acidic (H_2SO_4) and basic (NaOH) pH modifiers were obtained from Fisher Scientific Co.

In accordance with the FIPR requirement of maintaining the Ca^{+2} and Mg^{+2} levels in water at 40 ppm and 20 ppm respectively these ions were added as acetates. Gainesville tap water was found to contain 18±1.0 ppm Mg^{+2} and 27±1.0 ppm Ca^{+2}. 51.6 mg Ca-acetate and 16.08 mg Mg-acetate per liter of tap-water were added to achieve the desired water-quality.

Methods

Flotation: A schematic of the two-stage conditioning flotation process is presented in Figure 1. Flotation tests were carried out in a Denver model D-12 1.5 L laboratory flotation cell. Normally the conditioning pulp density of the 35x150 mesh feed is about 70%. However, in the present case a finer feed was used and to keep it in suspension more water was required. Consequently a pulp density of 61 wt % was maintained in the present tests during the first stage conditioning and 35-40 wt.% during the second conditioning stage. Flotation feed material was first conditioned at pH 10 with fatty acid followed by flotation of apatite and dolomite at pH 9-10. The float fraction was then re-conditioned at lower pH, preferably at a value of 4. As has been described in earlier work [2] frother addition was necessary during flotation at pH 5.0 or below due to the poor frothing characteristic of fatty acid collectors in the low pH range.

Analysis: The P_2O_5 and MgO content of various fractions were determined using acidic digestion followed by quantitative analysis using a Perkin Elmer Plasma II inductively coupled plasma emission spectrometer. The insolubles were determined gravimetrically.

RESULTS AND DISCUSSION

Details of the physicochemical mechanisms involved in selective dolomite flotation using the two-stage conditioning process have been described elsewhere [1]. An analysis of the adsorption of oleate on apatite and dolomite as a function of pH (Figure 2) reveals the underlying mechanisms [1]. As can be seen, oleate adsorption on dolomite at pH 10 or higher is more than that on apatite. When the suspension pH is lowered from 10 to 4 or below the amount of adsorbed oleate on both minerals remains unchanged. However, the nature of the adsorbed species changes from the more active oleate complex at pH 10 to the less effective oleic acid at pH 4. The difference in the flotation response between apatite and dolomite in the two-stage conditioning process, has been attributed to a combination of the larger quantity of oleate adsorbed on dolomite and to the lower effectiveness of the adsorbed species as collectors at low pH.

The effects of certain processing parameters such as pH, collector dosage and collector type on selective flotation of dolomite were evaluated in this study. A 30 s reconditioning time was determined to be optimum [2]. An increase in reconditioning time resulted in decreased selectivity in the dolomite flotation stage due to an increase in apatite flotation. Preliminary results (Table 2) showed that the optimum pH for second stage conditioning is 4. Under this condition, an apatite concentrate analyzing 25.07% P_2O_5, 0.94% MgO and 12.39% insol was obtained at a P_2O_5 recovery of 90.2%. This corresponds to a 26% reduction in MgO content. Dolomite flotation was suppressed when the conditioning pH was less than 4. As explained in earlier studies a decrease in reconditioning pH (i.e. from pH 4 to pH 3) is expected to increase the kinetics of oleic acid formation (pK_a oleic acid = 4.95) and, hence, result in decreased flotation of both apatite and dolomite. On the other hand, an increase in reconditioning pH to 4.5 resulted in relatively more apatite flotation than at pH 4 and hence the recovery of P_2O_5 decreased. Further tests were therefore conducted with reconditioning pH of 4.

Table 3 shows the effect of collector dosage on the selective flotation of dolomite when the reconditioning pH was 4. It can be observed that higher collector dosage results in a further reduction in MgO content. The optimum conditions for dolomite removal were determined to be reconditioning pH 4, reconditioning time 30 s, dolomite flotation at pH 5 and collector dosage of 2.5 kg/t. Under these conditions MgO content decreased by 43% from the deslimed value of 1.27%. On an insol-free basis the MgO content of the concentrate is 0.83% which is below the desired limit of 1% MgO. The P_2O_5 content of the concentrate analyzed 25.14% (54.9% BPL) at about 90% recovery level. On an insol-free basis the P_2O_5 content would be 29% (63.3% BPL).

The optimized conditions under which minimum MgO could be obtained in the concentrate were also applied to flotation of 35x150 mesh feed (1.47% MgO). The P_2O_5 recovery was reduced to 50%. The concentrate analyzed 24.9 % P_2O_5 and 1.10% MgO. It was not possible to optimize the processing parameters for 35x150 mesh sample due to the paucity of feed.

Batch flotation tests were also conducted using 50:50 fatty acid:fuel oil collector to reduce fatty acid consumption. Table 4 shows the effect of reconditioning pH on the selective flotation of dolomite. It is seen that when the reconditioning pH is 5 the MgO content is reduced to 0.74% in the concentrate. However, the P_2O_5 recovery was only 35%. A major fraction of the feed floated out when it was reconditioned at pH 5. The reason for this occurrence is that the kinetics of oleate to oleic acid conversion is sluggish at pH 5 and this was not compensated by increasing the reconditioning time to an optimal level. Further tests were conducted at a reconditioning pH of 4 as a function of collector dosage (Table 5). At a collector dosage of 2.5 kg/t the MgO content in the concentrate was reduced to 1.09% at P_2O_5 recovery of 90%. A comparison of the results presented in Table 5 with those in Table 3 shows that fatty acid alone is more effective in selectively floating out dolomite.

REFERENCES

1. Chanchani, R., "Selective Flotation of Dolomite from Apatite using Sodium Oleate as the Collector", PhD Thesis, University of Florida, 1984.

2. Moudgil, B.M., Ince, D., Vasudevan, T.V. and Sober, D.L., "Bench-scale optimization of the two-stage conditioning process for apatite-dolomite separation", Minerals and Metallurgical Processing, 1990, p. 53-56.

Table 1. Chemical analysis of as-received pebble fraction and prepared flotation feed.

Sample	Analysis, %					
	P_2O_5	CaO	Fe_2O_3	MgO	Al_2O_3	Insol
As-received (Pebble)	26.25	41.19	1.05	2.16	1.07	12.22
35x65 (Flotation Feed)	26.29	40.67	0.63	1.75	0.86	12.52
65x150 (Flotation Feed)	25.11	38.82	0.71	1.45	0.93	13.54
Deslimed Feed 65x150	24.55	36.73	0.74	1.27	0.82	14.32

Table 2. Effect of reconditioning pH on the selective flotation of dolomite with fatty-acid collector.

Collector dosage = 2.1 kg/t

Reconditioning pH	Grade % P_2O_5	% MgO in Concentrate	P_2O_5 Recovery %	Insolubles wt. %
3.5	25.52, 25.10	1.10, 1.08	96.4, 95.7	11.65, 12.77
4.0	25.07	0.94	90.2	12.39
4.5	24.94	1.06	85.3	12.71

Table 3. Effect of dosage of fatty-acid collector on the selective flotation of dolomite.

Reconditioning pH = 4
Flotation pH = 5

Collector Dosage kg/t	Grade % P_2O_5	% MgO in Concentrate		P_2O_5 Recovery %	Insolubles wt. %
		As is	Insol free basis		
1.8	24.84	1.25	1.43	84.4	12.43
2.0	24.28	1.06	1.19	92.7	11.10
2.1	24.68	1.21	1.42	96.4	14.50
2.4	25.14, 25.14	0.81, 0.84	0.93, 0.97	90.7, 95.0	13.28, 13.68
2.5	25.32, 25.06	0.64, 0.72	0.73, 0.83	89.3, 91.2	12.73, 13.28

Modifier Consumption

NaOH = 0.86 kg/t

H_2SO_4 = 0.60 kg/t

Table 4. Effect of reconditioning pH on the selective flotation of dolomite with fatty-acid-fuel oil collector.

Collector Dosage = 2 kg/t

Reconditioning pH	Grade % P_2O_5	% MgO in Concentrate	P_2O_5 Recovery %	Insolubles wt. %
3.0	25.00	1.22	90.37	12.71
3.5	24.83	1.16	95.00	12.67
4.0	25.01, 25.34	1.20, 1.32	86.58, 85.53	12.11, 11.80
4.5	25.54	1.18	61.87	11.07
5.0	24.74	0.74	34.8	13.38

Table 5. Effect of dosage of fatty-acid fuel oil collector on the selective flotation of dolomite.

Reconditioning pH = 4

Collector Dosage kg/t	Grade % P_2O_5	% MgO in Concentrate	P_2O_5 Recovery %	Insolubles wt. %
1.8	25.95	1.25	83.47	9.51
2.0	25.34	1.32	85.53	11.80
2.5	25.19	1.09	90.80	12.18

MINERAL RESOURCES INSTITUTE
COLLEGE OF ENGINEERING
THE UNIVERSITY OF ALABAMA
Box 870204
Tuscaloosa, AL 35487-0204

(205) 348-6577
FAX (205) 348-7612

FLORIDA INSTITUTE
OF
PHOSPHATE RESEARCH
1992 SEP -8 PM 1:25

September 1, 1992

Dr. Hassan El-Shall
Beneficiation Director
Florida Institute of Phosphate Research
1855 W. Main Street
Bartow, Florida 33830-7718

Dear Dr. El-Shall:

In response to your memorandum of August 24, 1992, the Mineral Resources Institute agrees to participate in the FIPR Program for evaluation of Dolomite Separation Techniques. As developers of the MRI "No-Conditioning Dolomite/Phosphate Flotation Process" we appreciate this opportunity very much and will make all efforts to offer you complete cooperation and support in achieving the goals of this project.

As discussed, over the telephone, you will ship a sample of about 200 pounds of high MgO pebble fraction (-5 + 16 mesh) to us at the Mineral Resource Institute. Upon receiving the sample, we will crush, grind, size and deslime at 150 mesh to prepare it for the required flotation feed. The testing program will include single or multi-factor factorially designed laboratory experiments to optimize the flotation separation conditions. The products of separation will be analyzed for MgO, P_2O_5 and acid insoluble to determine the quality and recovery of the final phosphate concentrate. The results of this limited investigation will be discussed and evaluated. We agree that the cost of performing the proposed work will not exceed $4,000. A final report including all the elements requested in your memorandum, will be submitted to FIPR on January 8, 1993.

We are looking forward to working with you and FIPR on this project. I wish you the best of luck in your new job with Allied Colloids. If you have any questions, please contact me. Best regards for you and the Family.

Sincerely,

John Hanna
Research Engineer

JH/skw
CC: Files

Approved Verle N. Schrodt
Dr. V.N. Schrodt
Assistant Dean
College of Engineering

FLOTATION OF GROUND HEAVY MEDIA DOLOMITIC PHOSPHATE PEBBLE REJECT

INTRODUCTION

The dolomite-apatite flotation process developed at the University of Alabama (U of A) in Tuscaloosa, Alabama, was evaluated on bench scale at the Florida Institute of Phosphate Research (FIPR). The test sample, which was provided by the then IMC Fertilizer, Inc. was pebble size heavy media reject material. Prior to flotation, the as received sample was ground to about minus 35 mesh and deslimed at 150 mesh. The 35 x 150 mesh fraction was then used as flotation feed. The testing procedure involved an initial "carbonate" flotation stage at pH 5.5-6.0, followed by a phosphate flotation stage at pH 6-7.

SUMMARY AND CONCLUSIONS

Batch rodmilling grinding for 15 minutes at 50% solids yielded a product that had only 45% of the original P_2O_5 in the flotation feed.

Carbonate and phosphate flotation, in Bartow city water, using the U of A procedure, produced froths without mineralization. Hence, flotation products could not be collected. and testing in deionized water had to be resorted to.

Although carbonate and phosphate flotation in deionized water yielded froths with significant mineralization, inadequate selectivity during the carbonate flotation stage precluded the production of low MgO phosphate concentrates during the phosphate flotation stage.

Thus, based on the test results shown in this report, the U of A dolomite-apatite flotation procedure may not be suitable for upgrading flotation feeds derived from comminuting pebble size material to minus 35 mesh.

It is envisaged that grinding to 100% minus 48 mesh and desliming at 325 mesh or finer, would help reduce phosphate concentrate MgO's to acceptable levels, and improve P_2O_5 recoveries .

SAMPLE TESTED

The test sample provided by IMC Fertilizer was heavy media reject material from its Four Corners plant. It was about 95% plus 16 mesh, and analyzed 25.9% P_2O_5, 2.7% Mgo and 12.3% acid insolubles (insol).

Grinding was done in 1000 gram batches for 15 minutes at 50% solids in a rubber-lined steel rodmill. The mill product was screened at 35 and 150 mesh as required by FIPR. Due to time constraints, the plus 35 mesh fraction, about 17% of mill product, was stored separately rather than reground with new feed. The minus 150 mesh fraction was also stored separately while the 35 x 150 mesh fraction was used as flotation feed. Size and chemical analyses of the mill product and flotation feed are shown in Tables 1 and 2, respectively.

TABLE 1. SIZE AND CHEMICAL ANALYSES OF MILL PRODUCT

Size Fraction Tyler Mesh	Weight %	Analysis %			%, Distribution		
		P_2O_5	MgO	Insol	P_2O_5	MgO	Insol
+35	17.2	26.70	1.45	14.88	17.7	9.4	20.4
35 x 150	46.0	25.47	1.89	16.10	45.3	32.3	59.1
-150	36.8	26.06	4.25	6.98	37.0	58.3	20.5
Feed	100.0	25.90	2.68	12.53	100.0	100.0	100.0

Conditions: 15 minutes rod-mill grind of a 1000 gm charge at 50% solids.

TABLE 2. SIZE AND CHEMICAL ANALYSIS OF FLOTATION FEED

Size Fraction Tyler Mesh	Weight %	Analysis %			%, Distribution		
		P_2O_5	MgO	Insol	P_2O_5	MgO	Insol
35 x 48	28.9	26.09	1.75	12.62	29.6	26.9	22.7
48 x 65	30.4	26.03	2.04	16.86	31.1	32.8	31.8
65 x 100	21.8	24.33	1.86	18.56	20.8	21.4	25.0
100 x 150	18.9	24.93	1.88	17.36	18.5	18.9	20.5
35 x 150	100.0	25.47	1.89	16.10	100.0	100.0	100.0

TEST PROCEDURE

Prior to flotation, the 35 x 150 mesh feed sample was "scrubbed" by agitation in the flotation machine at about 35% solids, for ten minutes, and rinsed on a 325 mesh screen.

Flotation tests were carried out in 250 gm batches using a Denver laboratory flotation machine with the impeller speed set at 900 rpm. The scrubbed material was pulped in the flotation cell at 16% solids using de-ionized (or tap) water. oleic acid (or Liqro GA) was used as collector and pine oil as frother.

Carbonate Flotation: The pulp pH was adjusted and maintained at the required pH level (5.5-6.0) by using dilute sulfuric acid. Thereafter, the fatty acid collector and frother were introduced into the pulp. Aeration of the pulp was begun, immediately after reagent addition, to float the carbonate gangue minerals.

Phosphate Flotation: The cell product. obtained from the carbonate flotation step was conditioned for three minutes with 0.5 lb./T. sodium silicate to depress siliceous gangue. This was followed by another two minute conditioning with the fatty acid collector. After collector conditioning, the pulp was again aerated to float the phosphate minerals leaving a silica rich tail in the cell product.

RESULTS

Agitation ("scrubbing") of the flotation feed at 35% solids prior to flotation produced slimes (minus 325 mesh) enriched in MgO and weighed about 4% of the flotation feed. The P_2O_5 analysis of these slimes was also slightly higher than that of the flotation feed. This indicates that some constituents of the phosphatic and dolomitic minerals tended to be fairly friable. Although they analyzed about 3.8% MgO on the average, MgO rejection in these slimes was below 10%.

Initial flotation testing, using 2 lb./T. collector (oleic acid or Liqro GA) in tap water (after scrubbing) produced poorly mineralized and unstable froths during carbonate flotation, and an almost completely barren but stable froth during the phosphate flotation step, as pH drifted higher than 6. This observation remained the same *even* after the collector dosage was tripled. A test result is shown in Table 3. Collector dispersion in the pulp was poor with or without emulsification with NaOH. The results shown in Table 3, for a test with 4 lb./T. Liqro GA, is typical of results obtained *at* lower or higher collector dosages, as well as with oleic acid.

subsequent flotation testing (of the "scrubbed" material) in de-ionized water, produced reasonably mineralized froths during the carbonate and phosphate flotation stages, although the carbonate flotation froth was not very stable. Some test results are shown in Table 4.

The first test in Table 4 shows the results of the "classical" no conditioning approach utilizing three carbonate flotation steps with 1 lb./T. oleic acid for each step, followed by phosphate flotation with 3 lb./T. oleic acid. The second test shows the results of an attempt to float the carbonates in one step with 4 lb./T. oleic acid, followed by a phosphate flotation step with another 4 lb./T. oleic acid.

For both tests, the final phosphate concentrates analyzed more than 30% P_2O_5 and about 1.5% MgO. The P_2O_5 recovery averaged 77.8 percent.

Testing with Liqro GA gave poorer results, due to incomplete carbonate flotation resulting from inadequate collector dispersion in the slightly acidic pulp. The Liqro GA, though emulsified with NaOH, was seen to recombine into small globs, soon after it was introduced into the pulp. It is possible that oleic acid had the same problem albeit to a lesser degree.

DISCUSSION

The u of A procedure for carbonate/phosphate flotation was originally developed on a feed consisting mostly of natural 35 x 150 mesh material, with a minimal quantity of ground pebble. The data presented in this report, indicate that it may not be suitable for upgrading 35 x 150 mesh high MgO feeds obtained solely from ground pebble reject. More importantly, the reason for the strong impact of water source on the process has to be thoroughly investigated.

At the time the procedure was developed in the 1980's, Tuscaloosa, Alabama city water, with a Ca^{++} content of about 14 ppm, was used for flotation. This may partly explain the difficulty of obtaining a mineralized froth in Bartow city water which had a Ca^{++} content of about 97 ppm, as determined by FIPR at the time of testing. Multivalent metal ions, such as calcium, are known to have a very deleterious effect on anionic collecting agents in general. Sigh solids conditioning of the feed with the anionic collector should ameliorate that deleterious effect. However, selectivity in carbonate/phosphate will be compromised--at least to some degree.

The use of deionized water for flotation helped restore froth mineralization in both flotation stages, but carbonate/phosphate separation was not selective enough to produce the low MgO phosphate concentrates desired. The reason for this may be found in Table 2 which showed that the MgO was coarsely segregated in the flotation feed. This point is particularly noteworthy because MgO was finely segregated in the U of A feed, and dolomite grains coarser than 65 mesh are known to be poorly floatable with anionic collectors, in slightly acidic media where chemisorption is not the predominant mode of collector adsorption.

FINAL REMARKS

Because the MgO grades of the phosphate concentrates produced are not considered optimum and are higher than metallurgically acceptable, a flowsheet has not been provided. Based on the above discussion, however, it is obvious that a flowsheet designed to successfully upgrade ground high MgO pebble, via anionic flotation of dolomitic gangue, should incorporate the following:

- stage grinding to 100% minus 48 mesh or finer to insure that dolomite grains are substantially minus 65 mesh.
- desliming at 325 mesh or finer to reduce P_2O_5 losses.
- high solids conditioning of the pulp with the anionic collector, for a short duration, to insure adequate dispersion of the "oily" collector and improve the chances for collector adsorption before contact with interfering cations in dilution water.

TABLE 3. FLOTATION OF GROUND HIGH MgO PHOSPHATE PEBBLE IN TAP WATER

Size Fraction Tyler Mesh	Weight %	Analysis %			%, Distribution		
		P_2O_5	MgO	Insol	P_2O_5	MgO	Insol
Scrub Slime	4.0	26.18	3.75	6.29	3.9	8.0	1.5
Dolo. Froth	4.8	26.80	4.55	4.85	4.8	11.6	1.3
Phos. Conc.	0.0	--	--	--	--	--	--
Cell Prod.	91.2	26.91	1.66	18.43	91.3	80.4	97.2
Composite	100.0	26.87	1.88	17.29	100.0	100.0	100.0

Test Conditions: Carbonate flotation for 1 min. with 4 lb./T. Liqro GA and 0.1 lb./T frother.
Phosphate flotation for 1 min., after conditioning with 0.5 lb./T. sodium silicate and additional 4 lb./T. Liqro GA.

TABLE 4. FLOTATION OF GROUND HIGH MgO PHOSPHATE PEBBLE IN DE-IONIZED WATER

Collector, lb./T.		Product	Weight %	Analysis %			%, Distribution		
Carb. Stage	Phos. Stage			P_2O_5	MgO	Insol	P_2O_5	MgO	Insol
3*	3	Scrub Slime	4.0	26.18	3.75	6.29	4.0	8.6	1.4
(OA)	(OA)	Dolo. Froth	6.0	23.65	7.14	5.60	5.5	24.5	1.9
		Phos. Conc.	63.3	30.89	1.45	5.19	75.4	52.5	18.1
		Cell Product	26.7	14.66	0.94	53.31	15.1	14.4	78.6
		Composite	100.0	25.93	1.75	18.11	100.0	100.0	100.0
4	4	Scrub Slimes	4.2	26.18	3.75	6.29	4.3	8.9	1.6
(OA)	(OA)	Dolo. Froth	7.7	24.30	5.77	3.09	7.3	25.2	1.4
		Phos. Conc.	68.2	30.49	1.54	3.70	81.0	59.5	15.2
		Cell Product	19.9	9.51	0.57	68.28	7.4	6.4	81.8
		Composite	100.0	25.66	1.77	16.61	100.0	100.0	100.0
4	4	Scrub Slimes	3.7	27.16	4.24	5.64	4.0	9.1	1.3
(LGA)	(LGA)	Dolo. Froth	3.5	23.85	4.99	3.67	3.3	10.1	0.8
		Phos. Conc.	77.6	29.06	1.74	5.02	89.4	78.0	23.3
		Cell Product	15.2	5.44	0.32	81.98	3.3	2.8	74.6
		Composite	100.0	25.22	1.73	16.69	100.0	100.0	100.0

* added in three doses of 1 lb./T. each
OA stands for oleic acid
LGA stands for Liqro GA

Frother dose - 0.06 lb./T for each carbonate flotation step.

United States Department of the Interior
BUREAU OF MINES
TUSCALOOSA RESEARCH CENTER
UNIVERSITY OF ALABAMA CAMPUS
P.O. BOX L
TUSCALOOSA, ALABAMA 35486-9777

June 15, 1993

FLORIDA INSTITUTE
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1993 JUN 16 AM 11:10

Dr. Richard F. McFarlin
Executive Director
Florida Institute of Phosphate Research
1855 West Main Street
Bartow, FL 33830-7718

Dear Dr. McFarlin:

Enclosed is the final report on the beneficiation study on high chromite ores.

If you have any questions, you can contact Brod Davis at (205) 759-9430 or Cy Jordan at (205) 759-9512.

Sincerely,

Bernard J. Scheiner
Supervisory Metallurgist

Enclosure

FINAL REPORT

FIPR CONTRACT ON RECOVERY OF PHOSPHATE FROM HIGH MGO ORE

INTRODUCTION

The Bureau of Mines and the Florida Institute of Phosphate Research (FIPR) agreed to cooperate on a research project designed to study the Bureau's existing technology for removal of MgO from Florida phosphate ores. FIPR provided the sample and requested that the Bureau (1) crush, grind, and screen the sample to fit the Bureau's process. (2) Run all of the necessary unit operations required using the Bureau's process to achieve optimum separation of MgO from the ore, together with optimum P_2O_5 recovery. (3) Report the flowsheet together with material balance. (4) Report reagent (types and amounts) additions, together with all other conditions of pH, pct solids, retention times, hydrodynamic conditions, etc. (5) Assay the feed and products by size fraction, together with recovery values. (6) List any environmental impact that could result due to the use of the Bureau's process.

The Bureau's approach involved grinding to obtain effective flotation size, scrubbing to liberate the soft dolomite, sizing to remove the fine liberated dolomite, and rougher flotation with fatty acid/fuel oil collector to recover the phosphate. An additional cleaner flotation step using sodium silicate was needed to produce an acceptable concentrate grade.

PROCESS FLOWSHEET

The proposed process flowsheet is shown in figure 1: The unit operations were each optimized to produce concentrates with good P_2O_5 grade and recovery and to reject the MgO containing dolomite. The pebble sample contained, in pct, 24.1 P_2O_5, 38.0 CaO, 1.85 MgO, 1.18 Al_2O_3, 1.12 Fe_2O_3, and 12.6 SiO_2. XRD analysis showed that the sample contained the minerals carbonate-fluorapatite, dolomite, and quartz. In this pebble sample, these minerals appeared to be mostly liberated, but were too coarse for efficient flotation. Preliminary flotation tests indicated that efficient flotation could be achieved with the material ground to pass 28 mesh. A hammermill grinder was chosen because it tended to minimize over-grinding which resulted in excessive loss of P_2O_5 to the fines. The hammermill was operated in closed circuit with a 28 mesh screen so that essentially all the flotation feed was minus 28 mesh. The ground material was sized at 150 mesh to remove fines produced during grinding.

It was determined that fines tended to be generated during conditioning and flotation of the material. Therefore, the ground material was scrubbed and sized prior to conditioning. Scrubbing was accomplished in a Denver laboratory scrubber at 50 pct solids for 20 min with 1.0 lb/ton of NaOH added for fines dispersion. The scrubbed material was sized at 150 mesh to remove fines generated during scrubbing. Table 1 shows the size analysis of the ground, scrubbed, and sized flotation feed. The table includes chemical analysis and distribution of the size fractions. Table 1 shows that the MgO content of the flotation feed was 0.82 pct, compared to 1.85 pct for the pebble. The reduction of MgO as a result of grinding, scrubbing, and sizing can be seen by comparing the MgO/P_2O_5 ratio of the pebble and flotation feed. The MgO/P_2O_5 ratio of the pebble was 0.077. The MgO/P_2O_5 ratio of the flotation feed was 0.037, indicating that the softer dolomite was preferentially removed by the grinding, scrubbing, and sizing. Table 2 shows the material balance for the process. The table shows that grinding, scrubbing, and sizing removed 58.8 pct of the MgO from the pebble, with a loss of 31.3 pct of the P_2O_5.

Conditioning was accomplished in a vertically stirred mixer at 72 pct solids. A typical fatty acid mixed with No. 5 fuel oil in a 2:3 ratio was the chosen collector. The amount of the mixed reagent used was 2.5 lb per ton of flotation feed. Conditioning time was 5 min at pH 9.2.

The conditioned feed was floated in a Denver laboratory flotation machine at 35 pct solids at pH 9.0 to 9.2. Since NaOH was used during scrubbing, no additional pH modifier was needed in the flotation step. Flotation was continued until the froth disappeared, usually less than 3 min. The phosphate rougher concentrate was subjected to a cleaner flotation with 1.0 lb/ton sodium silicate added. Water used in scrubbing, conditioning, and flotation was adjusted with calcium acetate and magnesium acetate to 40 ppm Ca and 20 ppm Mg to simulate flotation plant water. The phosphate concentrate produced contained, in pct, 29.6 P_2O_5, 46.4 CaO, 1.1 MgO, 1.32 Al_2O_3, 0.86 Fe_2O_3, and 7.2 SiO_2. Table 2 shows that 54 pct of the P_2O_5 contained in the pebble was recovered in the concentrate. The MgO/P_2O_5 ratio of the concentrate was 0.037, the same as for the flotation feed. It is apparent that the rougher and cleaner flotations essentially lowered the SiO_2 content. Table 3 shows other results from the flotation unit operation that could be achieved. The recoveries listed are from the flotation feed. The first data line in the table is for the proposed flowsheet. From the table, it is apparent that concentrates with lower MgO content can be produced, but at a sacrifice of P_2O_5 recovery. For example, lowering the reagent dosage *to 1.5* lb/ton resulted in a concentrate that contained 31.4 pct P_2O_5 and 0.96 MgO for a MgO/P_2O_5 ratio of 0.031. However, it resulted in an additional loss of 26 pct of the P_2O_5. Employing 3 cleaner flotations resulted in a concentrate containing 26.8 pct P_2O_5 and 0.69 pct MgO with a MgO/P_2O_5 ratio of 0.026.

However, an additional 7 pct of the P_2O_5 reported to the cleaner tailings. It is possible that this loss of P_2O_5 would be acceptable to make better grade.

OPERATING CONDITIONS

Listed below are the operating conditions for the proposed process.

Grinding: Hammermill in closed circuit with 28 mesh screen

Scrubbing: Denver scrubber, 1500 rpm, 50 pct solids, 20 min, 1.0 lb/ton NaOH

Conditioning: Vertical mixer, 500 rpm, 72 pct solids, 5 min, pH 9.2, 2.5 lb/ton fatty acid/fuel oil

Flotation: Denver cell, 1200 rpm, 35 pct solids, 3 min, pH 9.0 to 9.2, 6 L/min air flow rate

Cleaner flotation: 1.0 lb/ton sodium silicate

ENVIRONMENTAL IMPACT

It is anticipated that the proposed process would not result in any environmental impact that is different from current plant practices. Fines generated could be disposed in existing impoundments. Tailings are similar to those now produced and could be added to the general mill tailings. The three reagents used (fatty acid/fuel oil, NaOH, and sodium silicate) should not have any new environmental impact since they are already used in Florida phosphate flotation plants.

CONCLUSIONS

A phosphate pebble product that was high in MgO content was treated using a process previously devised by the Bureau of Mines. The pebble contained approximately 26 pct P_2O_5 and 2 pct MgO. By grinding, scrubbing, and sizing, followed by rougher and cleaner flotations, a concentrate was produced that contained 29.6 pct P_2O_5, and 1.1 pct MgO with an attendant P_2O_5 recovery of 54 pct of the P_2O_5 contained in the pebble product. A concentrate with lower MgO content could be produced by additional cleaner flotations, but with an accompanying loss of P_2O_5.

TABLE 1.--Size analysis of flotation feed

Screen size, mesh	Weight, pct	Chemical analysis, pct						Distribution, pct					
		P_2O_5	CaO	MgO	Al_2O_3	Fe_2O_3	SiO_2	P_2O_5	CaO	MgO	Al_2O_3	Fe_2O_3	SiO_2
28/35 . .	39.6	23.5	35.4	0.85	0.71	0.58	13.6	41.8	41.5	41.0	34.5	42.2	34.3
35/48 . .	28.4	20.1	30.9	.77	.59	.45	13.9	25.7	26.0	26.7	20.6	23.5	25.1
48/65 . .	12.5	22.2	33.1	.82	1.35	.53	19.7	12.4	12.2	12.5	20.7	12.1	15.6
65/100 .	9.4	21.7	33.1	.78	1.07	.53	20.6	9.2	9.2	9.0	12.4	9.2	12.4
100/150 .	6.5	23.3	35.4	.81	.91	.63	21.2	6.8	6.9	6.4	7.3	7.6	8.8
-150 . .	3.6	24.8	38.1	1.00	1.00	.81	16.4	4.1	4.1	4.4	4.5	5.4	3.8
Composite	100.0	22.2	33.7	.82	.81	.54	15.7	100.0	100.0	100.0	100.0	100.0	100.0

TABLE 2.--Material balance for proposed flowsheet

Product	Weight, pct	Chemical analysis, pct						Distribution, pct					
		P_2O_5	CaO	MgO	Al_2O_3	Fe_2O_3	SiO_2	P_2O_5	CaO	MgO	Al_2O_3	Fe_2O_3	SiO_2
Concentrate	43.1	29.6	46.4	1.10	1.32	0.86	7.2	54.0	52.3	26.8	44.6	40.7	18.7
C. tailings	5.0	21.6	37.7	2.55	1.24	0.76	22.0	4.6	4.9	7.2	4.9	4.2	6.6
R. tailings	17.1	14.0	22.7	0.75	1.11	0.47	55.2	10.1	10.2	7.2	14.9	8.8	56.8
Scrub fines	5.1	24.8	42.3	3.26	1.62	1.40	9.8	5.3	5.6	9.4	6.5	7.8	3.0
Grind fines	29.7	20.7	34.7	2.95	1.25	1.18	8.3	26.0	27.0	49.4	29.1	38.5	14.8
Composite .	100.0	23.6	38.2	1.77	1.27	.91	16.6	100.0	100.0	100.0	100.0	100.0	100.0

TABLE 3.--Flotation test results

Scrub time, min	Conditioning time, min	Reagent dosage, lb/ton	No. cleaner flotations	P_2O_5 grade, pct	P_2O_5 recovery, pct	MgO grade, pct
20	5	2.5	1	29.6	78.6	1.10
5	5	2.5	1	29.6	71.2	1.08
5,5	5	2.5	1	30.1	89.5	1.35
20	5	1.5	1	31.4	52.5	.96
20	5	2.0	1	29.9	80.5	1.17
20	5	2.5	3	26.8	71.8	.69

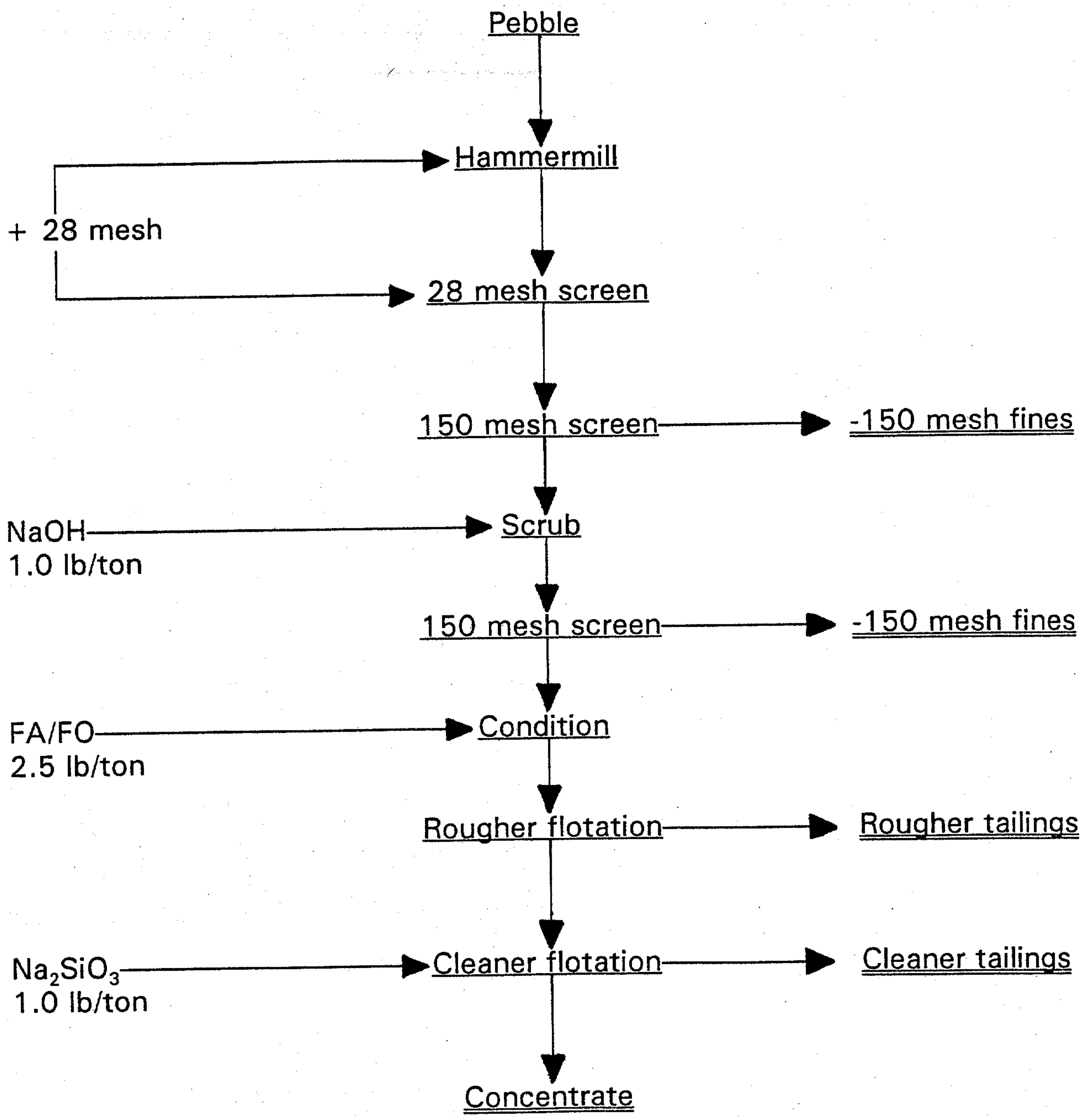


Figure 1 --Proposed flowsheet

TVA

Tennessee Valley Authority, Post Office Box 1010, Muscle Shoals, Alabama 35660

FLORIDA INSTITUTE
OF
PHOSPHATE RESEARCH
1993 MAY 19 AM 11: 24

May 17, 1993

Mr. Michael Bogan
Laboratory Director
Florida Institute for Phosphate Research
1855 West Main Street
Bartow, Florida 33830

Dear Mr. Bogan:

Enclosed is a report containing test results for the beneficiation of dolomitic phosphate pebble reject using the TVA diphosphonic depressant process. TVA will be billing FIPR $4,000 for this work. As agreed, TVA will cover the rest of the costs for this project.

As you requested, we are sending, under separate cover, a set of flotation product samples for verification of our analytical results. These samples include phosphate concentrate, dolomite float, silica float, and different size fractions of phosphate concentrate. The ground samples are the exact samples analyzed by our analytical laboratory.

If you have any questions, please do not hesitate to let me know. I may be reached at (205) 386-2770.

Sincerely,

Shuang-shi Hsieh

Shuang-shii Hsieh
Metallurgical Engineer
National Fertilizer and
Environmental Research Center

Enclosure

BENEFICIATION OF DOLOMITIC PHOSPHATE PEBBLE REJECT

By

S. S. Hsieh, J. C. Chowning, Jr., and J. Gautney

ABSTRACT

At the invitation of the Florida Institute of Phosphate Research (FIPR), TVA participated in a program to evaluate TVA's patented process for recovery of phosphate values from dolomitic phosphate pebble reject. The results of bench-scale flotation tests showed that TVA's diphosphonic depressant process can be used to beneficiate the dolomitic phosphate pebble submitted by FIPR. The process produced a phosphate product containing 30.7% P_2O_5, 1.01% MgO and 3.2% acid-insoluble matter using a -48 +400 mesh flotation feed. The P_2O_5 recovery was 92.7% from the deslimed flotation feed and 63.8% from the total pebble sample. The reagent dosages per metric ton were 0.3 kg diphosphonic acid reagent (60% active ingredient content), 0.6 kg oleic acid, 0.04 kg pine oil, and 0.3 kg dodecylamine hydrochloride. The reagents used in the TVA process should not cause any additional environmental problems. The process could also be easily adapted to current plants with the addition of a grinding and classification circuit.

INTRODUCTION

At the invitation of the Florida Institute of Phosphate Research (FIPR), the National Fertilizer and Environmental Research Center (NFERC), Tennessee Valley Authority (TVA), participated in a program to evaluate TVA's patented process for recovery of phosphate values from dolomitic phosphate pebble reject. FIPR provided the phosphate pebble reject sample and contributed $4,000 toward the expense of testing.

The pebble reject provided by FIPR was the heavy media float from Four Corners Mine. FIPR indicated that it was permissible for the investigators to crush, grind, and screen the reject to the size that best fit the investigator's own process. However, it was specified that the feed to the flotation process should not be less than 150 mesh.

The investigators were asked to perform the following tasks:

(1) Run all of the necessary unit operations required to achieve optimum separation of MgO from the pebble reject, together with optimum phosphate recovery, using the investigator's process.

(2) Provide a process flowsheet together with material balances.

(3) Report reagent added, together with all other conditions (pH, % solid, retention times, hydrodynamic conditions, etc.)

(4) Provide an assay of the feed and products by size fraction, together with recovery values.

(5) List any environmental impact that could result due to the use of the process.

(6) Provide any other information that would help FIPR in evaluating the process for commercial adaptation by the Florida phosphate industry.

(7) Provide FIPR with a sample of the products from the optimum (best) run for independent analyses by a reference laboratory.

EXPERIMENTAL

The dolomitic phosphate pebble sample used for the study was the heavy media float from Four Corners Mine. The sample contained 26.3% P_2O_5, 2.07% MgO, and 13.7% acid-insoluble matter. Detailed size and chemical analyses are shown in Table I.

The pebble sample was stage wet-ground in a 12" x 5" Denver laboratory ball mill to prepare a -48 +400 mesh flotation feed. The batch grinding time was 5 min and the circulating load was about 50%. The ground sample was screened with a 24" SWECO separator. The -400 mesh slime removed in this screening stage was 21.4% by weight of pebble sample (Figure 1).

The size and chemical analyses of the flotation feed before scrubbing and desliming are shown in Table II. The feed contained 25.7% P_2O_5, 1.50% MgO, and 16.2% acid-insoluble matter. This feed still contained 9.27% -400 mesh material. The MgO content in this -400 mesh fraction (2.99%) was higher than that in other size fractions. Excluding the -400 mesh material, the flotation feed contained 25.8% P_2O_5, 1.35% MgO, and 16.8% acid-insoluble matter (Table II). The -400 mesh slime from the preceding grinding and screeening stages was not analyzed, but its MgO content was estimated to be higher than 3%. The flotation feed was maintained in the wet state until used in the flotation tests. Before each flotation test, the flotation feed was scrubbed and screened to remove -400 mesh slime. This additional -400 mesh slime removed after scrubbing was about 7.3% by weight of the original pebble sample (Figure 1). Together with the weight loss in the previous grinding and screening, the total weight loss to the -400 mesh slime was about 28.7% of the original sample (Figure 1).

The equipment used for scrubbing, conditioning, and flotation was Denver model D-12 type. In the bench-scale tests, a 551-g sample (dry basis) was scrubbed at about 50% solid for 5 min and then screened to remove the -400 mesh slime fraction. The deslimed feed (about 500 g) then was conditioned at a pulp density of 65% solids for 1 min with diphosphonic acid reagent (60% active ingredient content) as a phosphate mineral depressant, and then for 2.5 min with oleic acid as a dolomite collector and pine oil as a frother. The pH was measured at the end of conditioning (no pH adjustment was performed). After conditioning, the pulp was transferred to the flotation cell and diluted with tap water (containing about 35 ppm Ca). The dolomite then was floated as the waste. The phosphate mineral and silica in the flotation cell were reconditioned with dodecylamine hydrochloride for 30 sec and silica was then floated as the waste. The phosphate mineral remained in the sink as the final product. All float and sink samples were filtered, dried, and then analyzed by NFERC's Chemical and Environmental Section (CEAS). The different size fractions of phosphate product were also analyzed as required by FIPR.

RESULTS AND DISCUSSION

The flowsheet used to process the dolomitic phosphate pebble reject is shown in Figure 1. As indicated above, the pebble sample was ground to -48 mesh and the total weight lost to the -400 mesh slime was 28.7%. Therefore, the material subjected to flotation was 71.3% by weight of the pebble reject. The material lost to slime was not analyzed, but estimated to contain 28.5% P_2O_5, 4.1% MgO, and 4.7% acid-insoluble matter (calculated from the data for the pebble sample in Table I: 26.3% P_2O_5, 2.07% MgO, and 13.7% acid-insoluble and the data for the flotation feed in Table III: 25.4% P_2O_5, 1.25% MgO, and 17.3% acid-insoluble matter). Dolomite particles in the pebble reject sample were soft, as expected, and disproportionally distributed in the -400 mesh fraction, after grinding.

Results from the best flotation test are present in Table III. The reagent dosages were 0.3 kg/t diphosphonic acid reagent (60% active ingredient content), 0.6 kg/t oleic acid, and 0.04 kg/t pine oil for dolomite flotation, and 0.3 kg/t dodecylamine hydrochloride for silica flotation. In this test, the phosphate product contained 30.7% P_2O_5, 1.01% MgO, and 3.2% acid-insoluble matter; the P_2O_5 recovery from the flotation was 92.7% and the weight recovery was 76.56%. When the loss of material in the slime was taken into account, the P_2O_5 recovery (from the original pebble sample) was 63.8% and the weight recovery was 54.6%. The dolomite float (7.21% by weight) contained 14.5% P_2O_5, 5.98% MgO, and 18.3% acid-insoluble matter. This indicates that dolomite was effectively removed using diphosphonic acid as a phosphate mineral depressant. The silica float (16.24% by weight) contained 5.0% P_2O_5, 0.27% MgO, and 83.6% acid-insoluble matter. Dodecylamine hydrochloride was used in this test to remove silica, but other types of amine reagents can be substituted depending on the preference of the individual phosphate company. The calculated composition of the deslimed flotation head sample was 25.4% P_2O_5, 1.25% MgO, and 17.3% acid-insoluble matter. The data were reasonably close to that of the -48 +400 mesh fraction of feed shown in Table II, for which the calculated compositions were 25.8% P_2O_5, 1.35% MgO, and 16.8% acid-insoluble matter.

The different size fractions of the phosphate product were also analyzed. The resulting analyses are shown in Table IV. The composition of the phosphate product calculated from the analyses for the different size fractions is 30.0% P_2O_5, 1.00% MgO, and 3.80% acid-insoluble matter. The -48 +65 mesh fraction contained 29.0% P_2O_5, 1.30% MgO, and 5.63% acid insoluble matter, and the -65+100 mesh fraction contained 29.4% P_2O_5, 1.22% MgO, and 3.80% acid-insoluble matter. All other finer size fractions contained more than 30% P_2O_5, less than 1.0% MgO, and less than 4.0% acid-insoluble matter. Therefore, 100 mesh was the effective size for liberation of dolomite from the phosphate mineral. However, for practical beneficiation, the -48 +400 mesh flotation feed can be used to produce a phosphate product containing about 30% P_2O_5 and 1% MgO.

The environmental impact due to the use of this process should not be unfavorable. The process will generate about 30% by weight of -400 mesh slime. However, this amount of slime is small compared to the amount of slime from the current phosphate mining operation. The reagents used in this process were fatty acids, amines, pine oil, and diphosphonic acid. The fatty acids and amines are used in the phosphate industry and pine oil is widely used as a frother in other mineral industries. As for diphosphonic acid, it is used for scale and corrosion control, chelation, and dispersion. This reagent is reportedly "slightly toxic" by ingestion, "practically non-toxic" by dermal application, "moderately irritating" to the skin, and "corrosive" to the eyes (Monsanto's Technical Bulletin No. IC/SCS-323). Therefore, the use of TVA's flotation process should not present any significant additional environmental problems for the phosphate industry.

The TVA process could easily be adapted to the current phosphate beneficiation processes. The rougher flotation circuit could be used for the dolomite flotation and the amine flotation circuit could be used to float silica. The only major additional equipment required would be the grinding and classification system, which is used for the preparation of the -48 +400 mesh flotation feed.

CONCLUSIONS

The flotation tests indicate that the TVA diphosphonic acid depressant process can be used to beneficiate the dolomitic phosphate pebble submitted by FIPR. In bench-scale tests, the process produced a phosphate product containing 30.7% P_2O_5, 1.01% MgO, and 3.2% acid-insoluble matter using a -48 +400 mesh flotation feed (Table III). The P_2O_5 recovery was 92.7% from the deslimed flotation feed and 63.8% from the total pebble sample. The weight recovery was 76.6% from the deslimed flotation feed and 54.6%. from the total pebble sample. The loss of pebble sample to -400 mesh slime due to grinding, classification, and desliming was 28.7%. The reagent dosages per metric ton of feed in the flotation were 0.3 kg diphosphonic acid reagent (60% active ingredient content), 0.6 kg oleic acid, 0.04 kg pine oil, and 0.3 kg dodecylamine hydrochloride. The reagents used in the TVA process should not cause any additional environmental problems. The process could also be easily adapted to current plants with the addition of a grinding and classification circuit.

Table I. Size and Chemical Analyses of Dolomitic Phosphate Pebble Reject from Heavy Media Float

Size, Mesh			Analysis, %			Distribution, %		
Passing	Retained	Wt.%	P_2O_5	MgO	A.I.[a]	P_2O_5	MgO	A.I.[a]
	6	3.81	25.5	3.08	11.3	3.7	5.7	3.2
6	8	20.62	26.0	3.38	8.8	20.4	33.7	13.2
8	10	24.37	27.1	2.07	11.2	25.1	24.3	20.0
10	14	31.53	26.0	1.51	17.3	31.2	23.1	40.0
14	20	17.05	26.6	1.23	16.3	17.2	10.2	20.4
20	-	2.62	24.7	2.32	17.3	2.5	2.9	3.2
Head[b]		100.00	26.3	2.07	13.7	100.1	99.9	100.0

---

[a] Acid-insoluble matter

[b] Calculated

Table II. Size and Chemical Analyses of -48 +400 Mesh Flotation Feed Before Scrubbing and Desliming

Size, Mesh			Analysis, %			Distribution, %		
Passing	Retained	Wt.%	P_2O_5	MgO	A.I.[a]	P_2O_5	MgO	A.I.[a]
48	65	12.01	23.8	1.20	21.4	11.1	9.6	15.9
65	100	23.55	25.9	1.37	16.8	23.7	21.6	24.5
100	150	20.28	25.7	1.28	17.5	20.3	17.3	22.0
150	200	17.62	26.1	1.36	15.4	17.9	16.0	16.8
200	270	10.44	26.6	1.41	15.2	10.8	9.8	9.8
270	400	6.83	26.8	1.57	13.2	7.1	7.2	5.6
400	-	9.27	25.5	2.99	9.5	9.2	18.5	5.4
Head[b]		100.00	25.7	1.50	16.2	100.1	100.0	100.0
48	400[b]	90.73	25.8	1.35	16.8	90.8	81.5	94.6

[a] Acid-insoluble matter

[b] Calculated

Table III. Results of Flotation Test with -48 +400 Mesh Feed

Reagent, kg/t					Product		Analysis, wt. %			Distribution, %		
Oleic Acid	Diphosphonic[a] Acid	Amine[b]	Pine Oil	Conditioning pH	Name	Wt. %	P_2O_5	MgO	A.I.[c]	P_2O_5	MgO	A.I.[c]
0.6	0.3	-	0.04	6.2	Dolomite Float	7.21	14.5	5.98	18.3	4.1	34.5	7.6
-	-	0.3	-	7.5	Silica Float	16.24	5.0	0.27	83.6	3.2	3.5	78.3
					Phosphate Sink	76.56	30.7[d]	1.01[d]	3.2[d]	92.7	62.0	14.1
					Head (calculated)	100.01	25.4	1.25	17.3	100.0	100.0	100.0

[a] Hydroxyethylidene diphosphonic acid, 60% active content

[b] Dodecylamine hydrochloride

[c] Acid-insoluble matter

[d] Average of chemical analyses of phosphate sink sample and its different size fraction samples (Table IV)

Table IV. Size and Chemical Analyses of Phosphate Sink (Concentrate)

Size, Mesh			Analysis, %			Distribution, %		
Passing	Retained	Wt.%	P_2O_5	MgO	A.I.[a]	P_2O_5	MgO	A.I.[a]
48	65	15.20	29.0	1.30	5.63	14.7	19.7	22.5
65	100	26.36	29.4	1.22	3.80	25.8	32.1	26.4
100	150	21.34	30.2	0.97	3.63	21.5	20.6	20.4
150	200	17.17	30.5	0.80	2.85	17.5	13.7	12.9
200	270	10.47	31.0	0.70	3.29	10.8	7.3	9.1
270	400	5.96	31.0	0.68	3.68	6.2	4.0	5.8
400	-	3.49	31.0	0.72	3.28	3.6	2.5	3.0
Head[b]		99.99	30.0	1.00	3.80	100.1	99.9	100.1

[a] Acid-insoluble matter
[b] Calculated

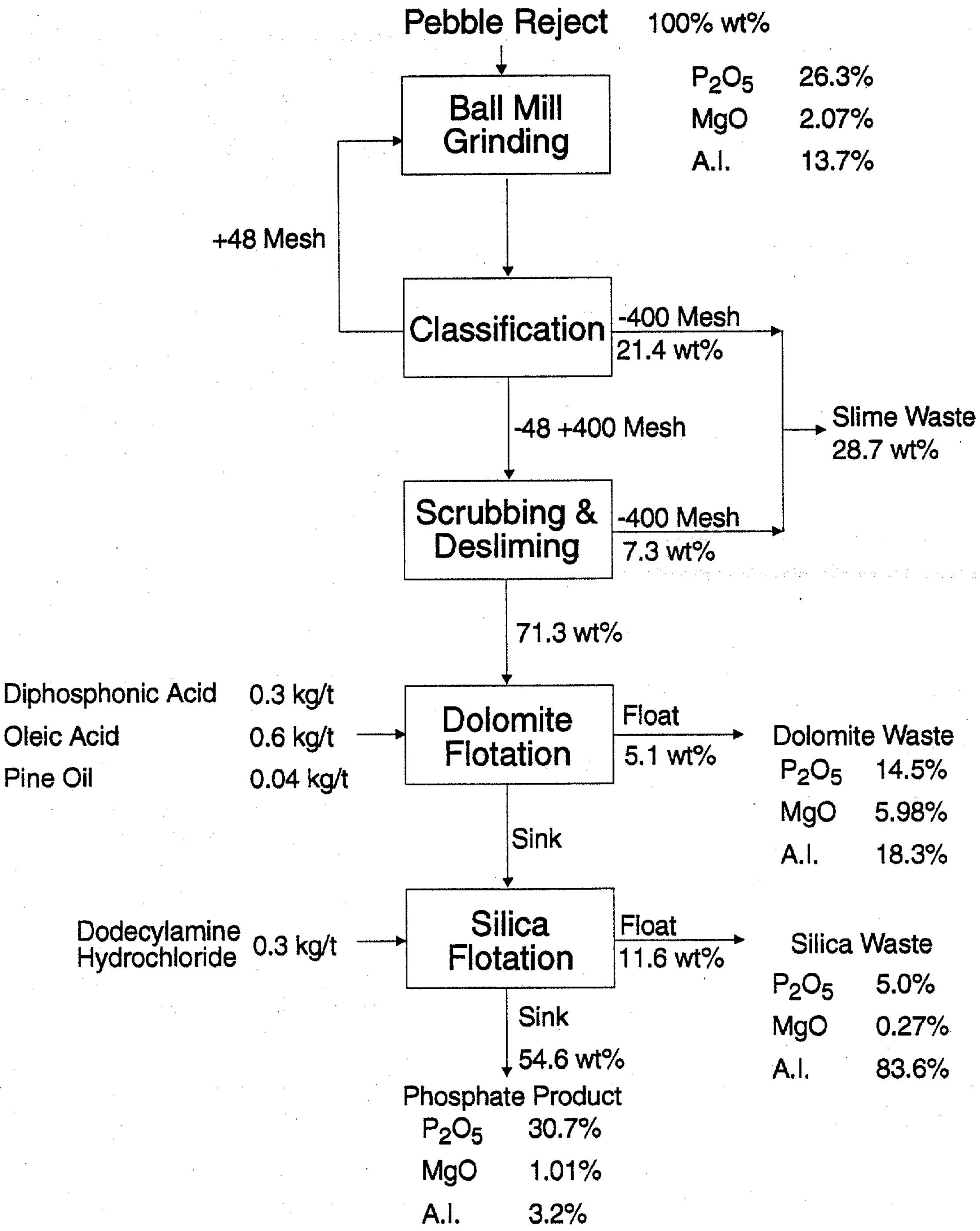


Figure 1. Simplied flowsheet for TVA's diphosphonic depressant process

APPENDIX B

GRINDABILITY CALCULATIONS

BALL MILL GRINDABILITY TEST

PURPOSE

To determine Materials Work Index according to the method developed by Mr. F.C. Bond.

COMMENTS

The experiment utilizes a 12" x 12" grindability mill which is equipped with a digital revolution counter and runs at 70 rpm.

The mill has a charge consisting of: 43 balls of 1.45" dia., 67 @ 1.17", 10 @ l.0", 71 @ 0.75", and 94 @ 0.50". Total charge weight is 20,125 grams with a calculated surface area of 842 sq. in.

PROCEDURE

1. Prepare -6 mesh material for work index determination.
2. Run a dry screen analysis on a feed sample at 8, 10, 14, 20, 28, 35, 48, 65, 100, and 150 mesh for 10 minutes on the Ro-Tap.
3. Determine the packed weight of 700 cc of the -6 mesh ore. This will be the weight fed to the grindability mill for each cycle to testing.
4. Grind the sample for 100 revolutions.
5. Empty the mill, place the media on a %-inch screen and brush clean, then return media to the mill.
6. Screen the sample at your assigned size (P_1) for 10 minutes on the Ro-Tap and weigh the products.
7. Calculate the crams of undersize produced per mill revolution. This is the G_{bp}. Remember to correct for the amount of undersize in the feed.
8. Calculate the weight of undersize that would correspond to a 250% circulating load at the charge weight you are using. This is done by dividing the feed weight by 3.5.
9. Return the screen oversize to the mill along with enough new feed to bring the charge to its original weight.
10. Grind for the number of revolutions necessary to produce a 250% circulating load according to your calculations in Step 8.

11. Repeat steps 5-10 until the net grams of undersize produced per revolution reaches equilibrium and reverses its direction of increase or decrease.

12. Screen analyze the undersize product from your final cycle and plot the results. P_{80} may be taken from this plot. F_{80} is taken from the screen analysis plot of the feed.

13. Calculate the work index according to the equation:

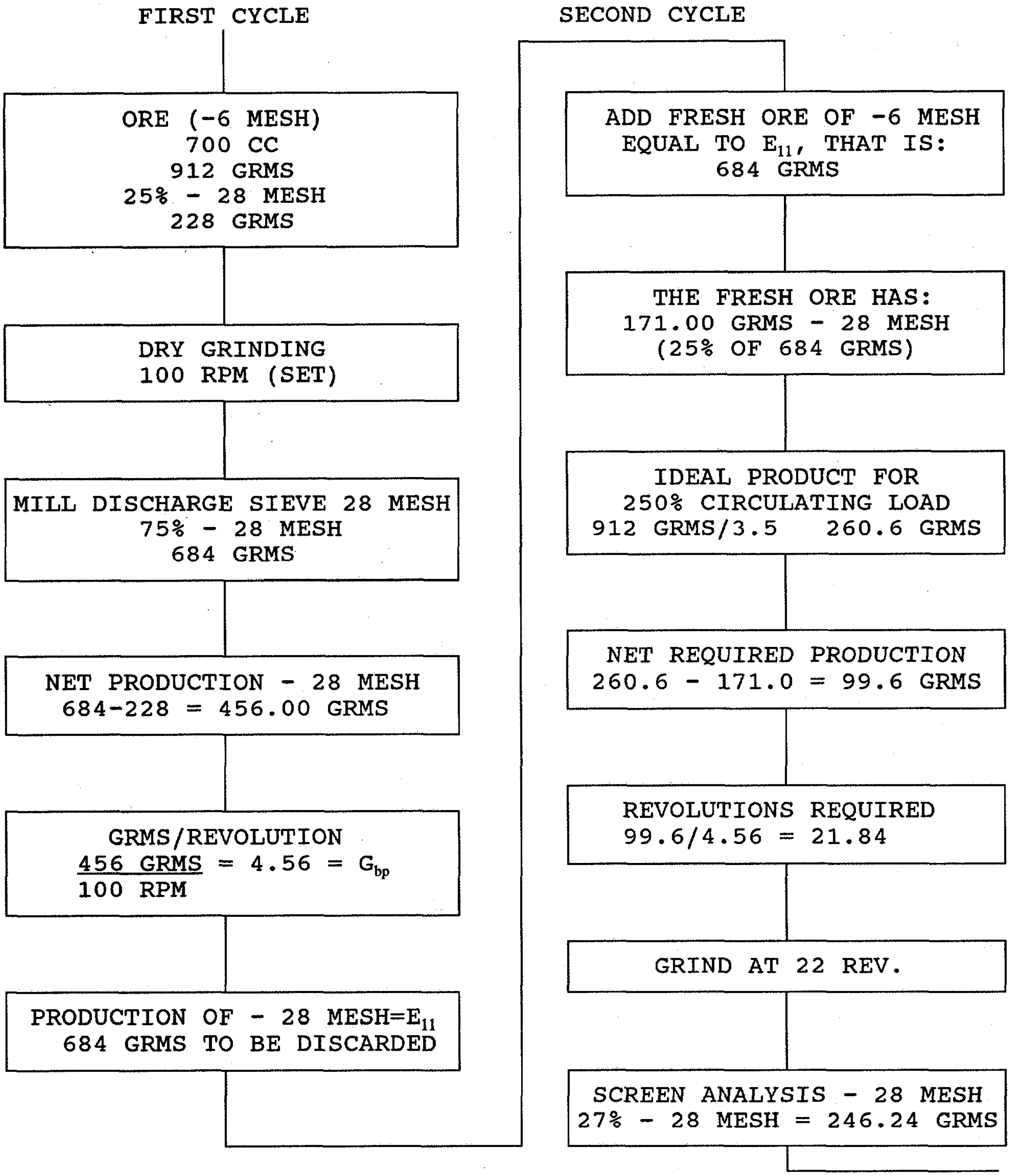
$$W = \frac{44.5}{(P_1)^{0.23} x (G_{bp})^{0.82} \left[\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}}\right]}$$

REFERENCES

1. Bond, F.C., Crushing and Grinding Calculations, Allis Chalmers Manufacturing Company, January 1962.

2. Smith, R.W., and K.H. Lee, A Comparison of Data From Bond Type Simulated Closed-Circuit and Batch Type Grindability Tests, AIME Transactions, Vol. 241, Society of Mining Engineers, March 1968.

GRINDABILITY TEST PROCEDURE
(Numerical Example)
(P_1 = 28 mesh)



CONTINUE...

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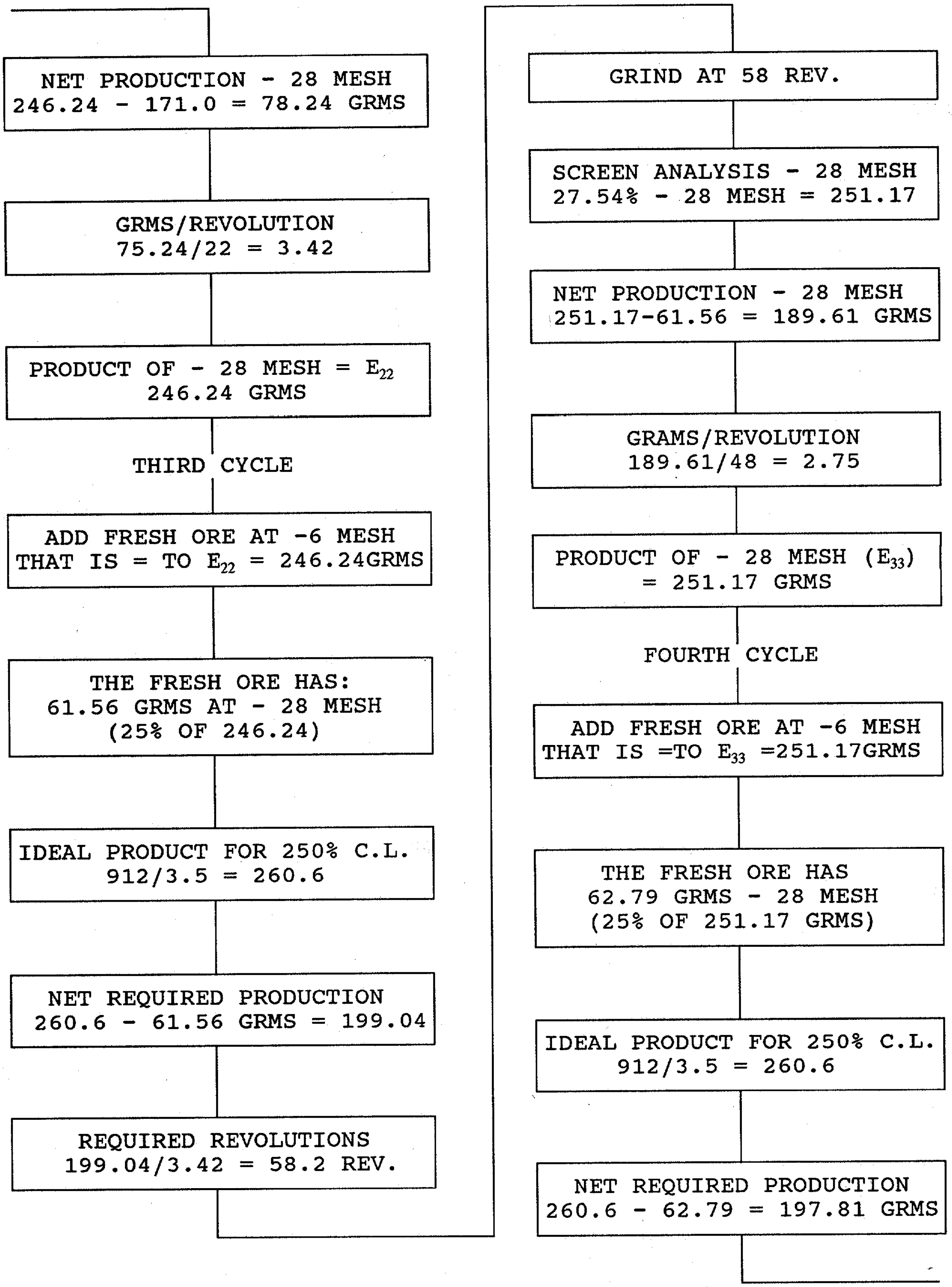

NET PRODUCTION - 28 MESH
246.24 - 171.0 = 78.24 GRMS

GRMS/REVOLUTION
75.24/22 = 3.42

PRODUCT OF - 28 MESH = E_{22}
246.24 GRMS

THIRD CYCLE

ADD FRESH ORE AT -6 MESH
THAT IS = TO E_{22} = 246.24GRMS

THE FRESH ORE HAS:
61.56 GRMS AT - 28 MESH
(25% OF 246.24)

IDEAL PRODUCT FOR 250% C.L.
912/3.5 = 260.6

NET REQUIRED PRODUCTION
260.6 - 61.56 GRMS = 199.04

REQUIRED REVOLUTIONS
199.04/3.42 = 58.2 REV.

GRIND AT 58 REV.

SCREEN ANALYSIS - 28 MESH
27.54% - 28 MESH = 251.17

NET PRODUCTION - 28 MESH
251.17-61.56 = 189.61 GRMS

GRAMS/REVOLUTION
189.61/48 = 2.75

PRODUCT OF - 28 MESH (E_{33})
= 251.17 GRMS

FOURTH CYCLE

ADD FRESH ORE AT -6 MESH
THAT IS =TO E_{33} =251.17GRMS

THE FRESH ORE HAS
62.79 GRMS - 28 MESH
(25% OF 251.17 GRMS)

IDEAL PRODUCT FOR 250% C.L.
912/3.5 = 260.6

NET REQUIRED PRODUCTION
260.6 - 62.79 = 197.81 GRMS

CONTINUE...

CONTINUED

REQUIRED REVOLUTIONS
197.81/2.75 = 71.9

GRIND AT 72 REV.

SCREEN ANALYSIS - 28 MESH
28% - 28 MESH = 255.36 GRMS

NET PRODUCTION - 28 MESH
255.36 - 62.79 = 192.57

GRMS/REVOLUTION
192.57/72 = 2.67

AND SO ON

<u>SUMMARY</u>

CYCLE	REVOLUTIONS	GRMS/REV (G_{bp})
1	100	4.56
2	32	4.13
3	48	3.95
4	50	3.85
5	51	3.84
6	51	3.83

Average = 3.84

REPLACING IN Eq. 1, ALL THE DATA COLLECTED WE GET:

$$\boxed{W = 12.36 \text{ kw-h/SHORT TON}}$$

DATA OF PHOSPHATE PEBBLES

1-Feed Size Distribution

Mesh	Cumulative % passing
6	94.2
8	71.0
10	45.5
12	26.1
14	12.3
20	0.7

By interpolation F_{80}=2600 Micron

2-Grinding Through 28 mesh(P_1=600 Micron)

2.1. Calculation of G_{bp}

CYCLE	REVOLUTIONS	GRMS/REV (G_{bp})
1	106	5.44
2	48	6.73
3	39	7.03
4	37	7.17
5	36	7.15
6	37	7.16

Average G_{bp}= 7.16

2.2 Product Size Distribution

Mesh	Cumulative % passing
28	100.0
35	74.6
48	55.3
65	41.9
100	31.0
150	22.6
200	14.7

By interpolation P_{80}= 475 Micron

2.3 Calculation of Work Index

Using the above parameter and Bond's Equation;

W_i = 7.7 KWH/Ton

3-Grinding Through 35 mesh(P_1=425 Micron)

3.1. Calculation of G_{bp}

CYCLE	REVOLUTIONS	GRMS/REV (G_{bp})
1	100	4.39
2	59	4.90
3	53	5.20
4	50	5.10
5	51	5.20
6	51	5.20

Average G_{bp}= 5.20

3.2 Product Size Distribution

Mesh	Cumulative % passing
35	100.0
48	69.4
65	50.6
100	36.5
150	26.1
200	16.2

By interpolation P_{80}= 325 Micron

3.3 Calculation of Work Index

Using the above parameter and Bond's Equation;

W_i = 8.0 KWH/Ton

4-Grinding Through 48 mesh(P_1=300 Micron)

4.1. Calculation of G_{bp}

CYCLE	REVOLUTIONS	GRMS/REV (G_{bp})
1	100	3.50
2	74	3.80
3	67	3.80
4	69	3.80
5	69	3.80
6	70	3.80

Average G_{bp}= 3.80

4.2 Product Size Distribution

Mesh	Cumulative % passing
48	100.0
65	71.4
100	49.5
150	34.9
200	17.9

By interpolation P_{80}= 220 Micron

4.3 Calculation of Work Index

Using the above parameter and Bond's Equation;

W_i = 8.4 KWH/Ton