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FLOTATION OF CARBONATE AND SILICATE MINERALS FROM PARTIALLY ALTERED
PHOSPHATE ROCK OF THE PHOSPHORIA FORMATION

by

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ABSTRACT

As part of its goal of helping to maintain an adequate supply of minerals to meet national economic and strategic needs, the Bureau of Mines, U.S. Department of the Interior, is conducting research to define the chemical and geological properties of complex low-grade phosphate rock occurring in the Phosphoria Formation and to develop methods for recovering phosphate values. The rock, which ranges from soft, friable shale to hard, tightly cemented matrix, is characterized as altered, partially altered, or unaltered. A flotation procedure currently under development for beneficiation of partially altered rock uses H_2SiF_6 to depress phosphate minerals, an anionic fatty acid collector for carbonate flotation, and cationic amine collectors for silica flotation. The procedure has been applied to several different rock types, and concentrates have been produced that range from 26 to 31 pct P_2O_5 with 57 to 85 pct P_2O_5 recovery.

INTRODUCTION

The United States has approximately 12 pct of the world phosphate reserves; about 31 pct of these are located in the Phosphoria Formation of the western phosphate field, which supplies about 12 pct of the U.S. phosphate rock production (1)*. Increasing demand for phosphate products indicates a small but continual growth in the western phosphate market. Current practice involves mining only the higher grade phosphate beds and stockpiling or leaving in place the lower grade materials that constitute the bulk of the potential reserves. As part of its effort to maximize minerals and metals recovery from domestic resources, the Federal Bureau of Mines is developing beneficiation and recovery systems for the complex lower grade phosphate materials to insure orderly future development of the western resources.

In past research the Bureau of Mines has made detailed studies on mining, beneficiating, processing, and marketing of western phosphate (2-6). Mable and Hess (7) conducted mineralogical and petrographic studies to classify the western phosphate ores. Additional beneficiation studies on various intermediate- and marginal-grade altered ores showed that phosphate could be concentrated by a combination scrubbing-sizing-flotation process (8,9). Current Bureau of Mines research on western phosphates was discussed in a recent article (10).

Past research was directed at utilization of intermediate- and marginal-grade ores (24 to 28 pct P_2O_5). Generally, a grade of 24 to 26 pct P_2O_5 is required for electric furnace processing, and a grade of 30 to 32 pct P_2O_5 is required for wet-process phosphoric acid production. There are sufficient high-grade reserves to meet present demands in the West. However, low-grade materials must be removed to reach high-grade beds, and it is expected that

*Underlined numbers in parentheses refer to items in the list of references at the end of this report.

these low-grade resources (10 to 24 pct P_2O_5) will be utilized in the future. Their utilization would mean longer mine life, conservation of resources, and less land disturbed by mining. The present research at the Bureau's Albany Metallurgy Research Center was undertaken to study and develop methods for producing acceptable concentrates from complex low-grade ores.

ACKNOWLEDGEMENT

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CHARACTERIZATION

Low-grade western phosphates occurring in the Phosphoria Formation vary considerably depending on location, depth of deposit, and degree of alteration resulting from weathering by surface exposure and ground water. In general, the phosphate minerals occur as pellets ranging in size from 106 to 600 μm^{**} , while the cementing carbonate and silicate gangue minerals generally have a much finer grain size. In highly altered materials the phosphate pellets are loosely cemented, and considerable upgrading is achieved by scrubbing and sizing. This is basically the material and the process used by the industry today. As the degree of alteration decreases, the degree of concentration attainable by scrubbing and sizing also decreases.

In addition to an increase in the gangue to pellet ratio, the quality of the pellets decreases in the low-grade materials. Gangue present as inclusions inside the pellets limits the apparent maximum grade attainable by conventional beneficiation.

Figures 1-3 show P_2O_5 analyses and cumulative weight percent and P_2O_5 distributions for typical altered, partially altered, and unaltered ores. For an altered ore, described in figure 1, over 50 pct of the weight can be removed as a low-grade slime tailings (minus 53 μm). The concentrate is significantly upgraded, often to acid grade (30 to 32 pct P_2O_5), and recovery of P_2O_5 is nearly 80 pct. A partially altered ore, described in figure 2, also can be upgraded by removal of a low-grade slime. Sizing at 53 μm removes about 30 pct of the weight, while over 80 pct of the P_2O_5 remains in the coarse fraction. Further beneficiation is required to produce an acid-grade concentrate. For an unaltered ore, described in figure 3, a large percentage of the weight is in the coarse sizes, and an acid-grade concentrate cannot be produced by sizing. The cumulative weight and cumulative P_2O_5 distribution curves nearly parallel one another, which means that no benefit is derived by size separation because gangue and P_2O_5 are removed in the same proportion. Some pellet liberation is obtained by scrubbing and sizing, but these liberated pellets are coated by calcite and silicate gangue constituents. Complete liberation is achieved only with extremely fine grinding. Thus, in flotation of unaltered materials, one must treat a partially liberated flotation feed to avoid the loss of selectivity that results from fine grinding.

Attempts to induce alteration by physical and/or chemical treatment generally have been unsuccessful. For calcareous unaltered phosphates, calcination followed by hydration and removal of lime by scrubbing results in considerable upgrading of the phosphate (11). However, this is a costly process that has high energy demands, and it is not applicable to unaltered siliceous materials.

**All size designations in this report conform to USA sieve series - ASTM specification E-11-70.

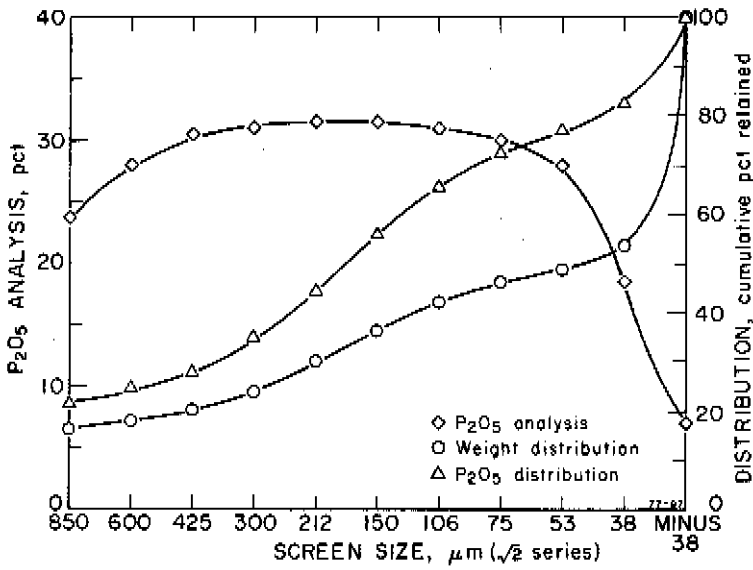


FIGURE 1 - Characterization curves for altered phosphatic shale

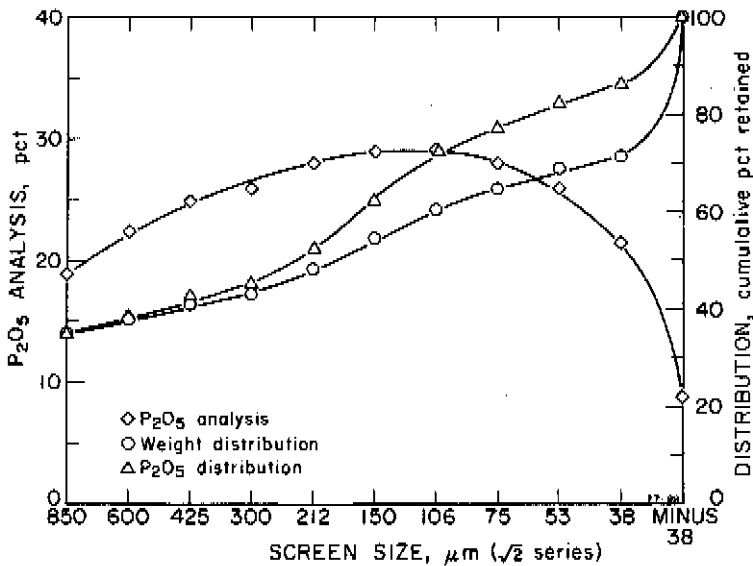


FIGURE 2 - Characterization curves for partially altered phosphatic shale

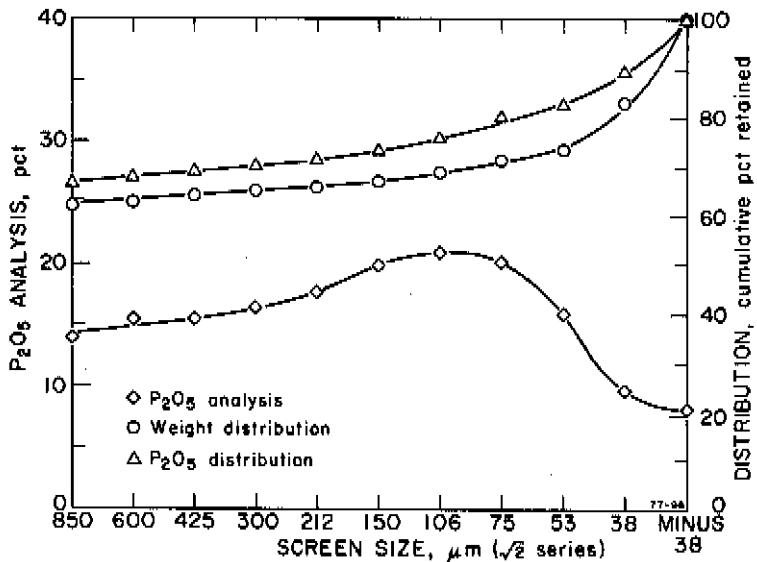


FIGURE 3 - Characterization curves for unaltered phosphatic shale

FLOTATION STUDIES

In a previous investigation (12), a flotation procedure was devised that employs H_2SiF_6 to depress phosphate minerals and a tall oil fatty acid-pine oil emulsion to float carbonate minerals from phosphate minerals. H_2SiF_6 is chemisorbed at the surface of the phosphate minerals, rendering them hydrophilic and preventing adsorption of collector. Under these conditions the fatty acid emulsion is a selective collector for the carbonate minerals. With mixed carbonate-silicate gangue most of the silica is depressed with the phosphate. Recent investigations showed that after carbonate flotation, silica can be removed by flotation with a cationic amine reagent. The amine can be added directly after carbonate flotation without any additional preparation of the pulp. Tests on a wide variety of materials showed that this procedure was effective if the phosphate could be liberated from the gangue minerals. The general test procedure for carbonate-silica flotation is shown in figure 4. A 1-kg sample of minus 12.5-mm material is attritioned at 65 pct solids in a blade attritioner and then screened to separate a flotation feed product, a coarse fraction, and a primary slime fraction. The coarse fraction is stage-ground in a laboratory rodmill at about 50 pct solids, and the ground product is combined with the flotation feed. Secondary slimes generated during grinding may or may not be included. The flotation feed is pulped to 20 to 25 pct solids and conditioned with H_2SiF_6 and the fatty acid-pine oil emulsion. Carbonate minerals are floated, and then the nonfloat fraction is conditioned with an amine solution made from equal parts of tallow and coco amine acetates. Silica is floated, and the remaining nonfloat product is the phosphate concentrate.

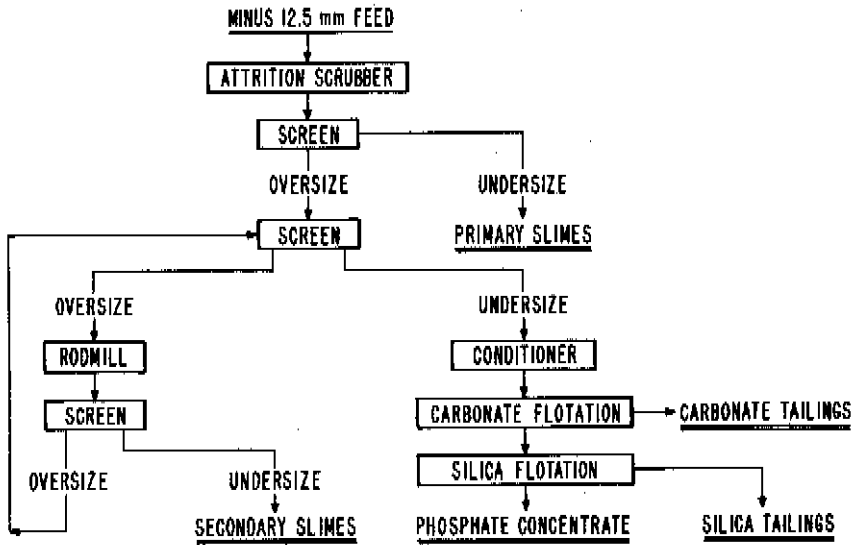


FIGURE 4 - General carbonate-silica flotation procedure

In some instances, dolomite can be activated with sodium metasilicate and then floated with amines (13). In tests where sodium metasilicate was used, it was added following carbonate flotation, and a short conditioning time was allowed before the amine solution was added. Results were inconclusive; the method seemed to help in some tests but not in others.

The carbonate-silica flotation method is still under development, and the test procedure is modified by addition or deletion of operations to adapt to the wide variety of low-grade deposits being studied. This report describes application of the method to four low-grade phosphatic shale samples from three different areas of Idaho.

Phosphatic Shale - Area A

The Area A sample was a low-grade, partially altered phosphatic shale from the lower shale section of the footwall at an operating mine near Soda Springs, Idaho. This sample had a typical analysis of 25.2 pct P_2O_5 , 39.9 pct CaO, 15.3 pct SiO_2 , and 1.07 pct MgO, and consisted of fine black phosphate pellets in a quartz-(Ca, Mg) CO_3 gangue matrix. There is some liberation at 425 μ m, but liberation is not complete even at 53 μ m.

Beneficiation schemes including sizing, flotation, and combinations of both were investigated. Results of an attritioning-grinding-sizing test indicated that a concentrate sized at 600 and 38 μ m would contain 29.3 pct P_2O_5 , 11.2 pct SiO_2 , and 0.8 pct MgO, with 80 pct P_2O_5 recovery. In a combination sizing-flotation scheme, a high-grade concentrate was separated at minus 600 plus 106 μ m by sizing. The remaining product was deslimed at 25 μ m and treated by carbonate and silica flotation. The combined concentrate from

this sizing-flotation procedure contained 30.2 pct P_2O_5 , 10.0 pct SiO_2 , and 0.7 pct MgO , and P_2O_5 recovery was 85 pct. Application of the general carbonate-silica flotation procedure shown in figure 4 to the Area A sample resulted in a concentrate that contained 31.2 pct P_2O_5 , 8.3 pct SiO_2 , and 0.6 pct MgO , with a P_2O_5 recovery of 82 pct. Results of this flotation test are shown in table 1. A comparison of the three beneficiation methods, table 2, shows that samples from this area can be beneficiated by the three schemes. A higher grade concentrate was achieved using the carbonate-silica flotation scheme.

TABLE 1 - Carbonate-silica flotation on deslimed lower shale - Area A

Product	Weight, percent	Metallurgical Data								CaO/ P_2O_5 ratio
		Analyses, percent				Distribution, percent				
		P_2O_5	CaO	SiO_2	MgO	P_2O_5	CaO	SiO_2	MgO	
Phosphate concentrate ^{1/}	68.4	31.2	47.6	8.3	0.61	82.2	80.5	40.7	49.3	1.53
Carbonate float	2.3	18.1	31.4	22.6	1.53	1.6	1.8	3.7	4.1	1.73
Rougher silica float	2.4	15.0	23.5	26.2	.91	1.4	1.4	4.5	2.6	1.57
Scavenger silica float	1.9	16.9	26.5	25.0	1.08	1.2	1.2	3.4	2.5	1.57
Primary minus 25 μ m slimes	18.0	12.5	21.9	28.4	1.18	8.7	9.7	36.7	25.1	1.75
Secondary minus 25 μ m slimes	7.0	18.4	30.9	21.8	1.99	4.9	5.4	11.0	16.4	1.68
Composite/totals	100.0	26.0	40.5	13.9	.85	100.0	100.0	100.0	100.0	1.56

Operating Conditions

Operation	Conditions		Reagents, kg/t ore				
	Time, minutes	pH ^{2/}	H_2SiF_6	NaOH	Fatty acid ^{3/}	Amine acetate ^{4/}	Na metasilicate
Condition	2		0.3	0.30			
Condition	3	7.2			0.25		
Carbonate float	2						
Condition	.5						0.13
Condition	1	7.9				0.25	
Rougher silica float	3						
Condition	.5						0.13
Condition	1	8.4				0.25	
Scavenger silica float	3						

^{1/} 0.59 pct Al_2O_3 , 0.85 pct Fe_2O_3 , 18.8 pct LOI at 790° C.

^{2/} Natural pH = 7.9.

^{3/} Emulsion: 15 g tall oil fatty acid, 0.2 g pine oil, 0.2 g NaOH, 285 ml H_2O .

^{4/} Solution: 1 pct coco amine acetate, 1 pct tallow amine acetate.

TABLE 2 - Comparison of beneficiation methods for lower shale - Area A

Method	Phosphate concentrate				
	P_2O_5	SiO_2	MgO	CaO/ P_2O_5	P_2O_5 recovery, pct.
Carbonate-silica flotation	31.2	8.3	0.6	1.53	82
Sizing-carbonate-silica flotation	30.2	10.0	.7	1.54	85
Attritioning-grinding-sizing	29.3	11.2	.8	1.55	80

Phosphatic Shale - Area B

The Area B sample represents another lower shale bed from present mining operations near Soda Springs, Idaho. In this sample, fine collophane pellets are contained in the partially altered dolomitic shale. Typical analysis is 18.8 pct P_2O_5 , 36.9 pct CaO, 18.7 pct SiO_2 , 3.83 pct MgO, and 11.8 pct loss on ignition at 790° C. Concentration by attritioning and sizing was not possible with this sample because of the finer pellet structure and a more tightly cemented matrix. A low-grade slime was removed, but the remaining sized product required further treatment. The very fine pellet-gangue locking occurring in this sample limits the maximum concentrate grade attainable. In the best carbonate-silica flotation test, the phosphate concentrate contained 26.2 pct P_2O_5 , 9.8 pct SiO_2 , and 2.5 pct MgO, with 66 pct P_2O_5 recovery and 14 pct loss on ignition at 790° C. Results are shown in table 3.

TABLE 3 - Carbonate-silica flotation on deslimed lower shale - - Area B

Product	Weight, percent	Analyses, percent				Distribution, percent				CaO/ P_2O_5 ratio
		P_2O_5	CaO	SiO_2	MgO	P_2O_5	CaO	SiO_2	MgO	
Phosphate concentrate ^{1/}	49.2	26.2	43.9	9.8	2.50	66.1	57.7	26.6	32.1	1.68
Rougher carbonate float	5.4	12.8	42.2	13.2	9.02	3.5	6.1	3.9	12.7	3.30
Scavenger carbonate float	5.2	14.5	42.3	13.2	7.39	3.9	5.9	3.8	10.0	2.92
Rougher silica float	2.2	12.1	22.8	32.2	3.37	1.4	1.3	3.9	1.9	1.88
Scavenger silica float	6.4	15.4	29.2	31.2	3.88	5.0	5.0	11.0	6.5	1.90
Primary minus 25 μ m slimes	16.3	12.3	25.7	28.3	4.19	10.3	11.2	25.5	17.9	2.09
Secondary minus 25 μ m slimes	12.7	13.7	33.9	21.5	5.24	8.9	11.5	15.1	17.4	2.47
Plus 212 μ m reject	2.6	6.9	19.4	71.3	2.17	.9	1.3	10.2	1.5	2.81
Composite/totals	100.0	19.5	37.4	18.1	3.83	100.0	100.0	100.0	100.0	1.92

Operating Conditions

Operation	Conditions		Reagents, kg/t ore			
	Time, minutes	pH ^{2/}	H_2SiF_6	Fatty acid ^{3/}	Amine acetate ^{4/}	Frother ^{5/}
Condition	4	5.9	0.38	0.50		
Rougher carbonate float	2					
Condition	2	6.7		0.25		
Scavenger carbonate float	2					
Condition	1	7.2			0.36	
Rougher silica float	3					
Condition	1	7.4			0.05	0.05
Scavenger silica float	3					

1/ 0.71 pct Al_2O_3 , 1.06 pct Fe_2O_3 , 13.8 pct LOI at 790° C.

2/ Natural pH = 8.0.

3/ Emulsion: 15 g tall oil fatty acid, 0.2 g pine oil, 0.2 g NaOH, 285 ml H_2O .

4/ Solution: 1 pct coco amine acetate, 1 pct tallow amine acetate.

5/ Polyglycol ether.

The MgO content of the concentrate has not been lower than 1.9 pct in any test. These results indicate that dolomite is contained in the phosphate pellets, or that Mg has replaced Ca in the collophane mineral lattice. A MgO level of about 2 pct appears to be the limit attainable with the carbonate-silica flotation scheme with acceptable P_2O_5 recovery. The Bureau of Mines has done some research on selective removal of magnesium impurities from phosphate concentrates by aqueous leaching with SO_2 (14), but more research is necessary on removal of MgO from these highly dolomitic phosphate materials.

Phosphatic Shale - Area C

In Area C near Pocatello, Idaho, low-grade phosphatic shale is removed during mining of the higher grade phosphate ores, which are utilized in production of fertilizer and elemental phosphorus. Stockpiled low-grade phosphatic shale in this area presently contains about 30 million tons of material. Altered, partially altered, and unaltered phosphatic shales are present in the stockpile. Samples of these three types obtained for study showed that in the altered shale phosphate pellets are liberated at a particle size of 150 μm , while the unaltered shale requires grinding to minus 75 μm for liberation. The partially altered sample was more friable than the unaltered material, and partial phosphate pellet liberation was achieved at minus 212 μm by controlled rodmill grinding.

The combination carbonate-silica flotation procedure was applied to a sample of the partially altered shale. From material that typically contained 19.4 pct P_2O_5 , 33.7 pct CaO, 22.8 pct SiO_2 , and 2.65 pct MgO, a concentrate was produced with a recovery of 57 pct of the P_2O_5 at a grade of 30.5 pct P_2O_5 , 6.9 pct SiO_2 , and 1.5 pct MgO. Results are shown in table 4.

TABLE 4 - Carbonate-silica flotation of partially altered shale - Area C

Product	Weight, percent	Metallurgical Data										
		Analyses, percent					Distribution, percent					CaO/ P_2O_5 ratio
		P_2O_5	CaO	SiO_2	MgO	LOI ^{1/}	P_2O_5	CaO	SiO_2	MgO		
Phosphate concentrate ^{2/}	37.4	30.5	47.0	6.9	1.49	11.3	56.9	50.4	11.2	20.4	1.54	
Carbonate float	2.1	17.7	36.8	18.2	4.36	25.4	1.9	2.2	1.7	3.4	2.07	
Silica float	23.9	18.7	35.0	23.2	3.73	16.2	22.3	24.4	24.0	32.7	1.87	
Plus 212 μm oversize	5.4	12.7	43.3	9.3	10.8	29.8	3.4	6.7	2.2	21.4	3.41	
Attrition slime (Minus 38 μm)	<u>31.2</u>	<u>10.0</u>	<u>18.2</u>	<u>45.0</u>	<u>1.93</u>	<u>20.5</u>	<u>15.5</u>	<u>16.3</u>	<u>60.9</u>	<u>22.1</u>	<u>1.82</u>	
Composite/totals	100.0	20.1	34.8	23.0	2.73	16.6	100.0	100.0	100.0	100.0	1.73	

Operating Conditions

Operation	Conditions		Reagents, kg/t ore				
	Time, minutes	pH	H_2SiF_6	NaOH	Fatty acid ^{3/}	Amine acetate ^{4/}	Amine ^{5/}
Condition	2		0.38				
Condition	5	7.4			0.75		
Carbonate flotation	5						
Condition	5	8.4		0.03		0.33	0.5
Silica flotation	8 est.					0.18	

^{1/} LOI at 1000° C.

^{2/} 0.56 pct Al_2O_3 , 0.92 pct Fe_2O_3 , 9.05 pct LOI at 790° C.

^{3/} Emulsion: 15.0 g tall oil fatty acid, 0.2 g pine oil, 0.2 g NaOH, 285 ml H_2O .

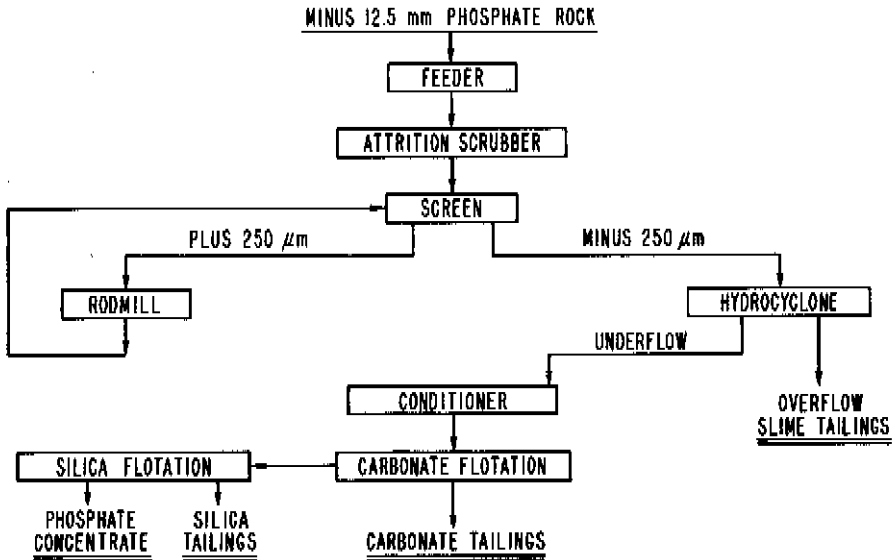
^{4/} Solution: 1 pct coco amine acetate.

^{5/} Tallow amine in 1:1 H_2O -ethanol solution.

CONTINUOUS-CIRCUIT BENEFICIATION

A 30-kg/hr continuous circuit was set up for evaluation of carbonate-silica flotation procedures. The flowsheet used in applying this flotation process to a partially altered phosphate rock is shown in figure 5. In this system minus 12.5-mm material was fed by a weigh feeder into a 4-liter blade attritioner operating at 570 rpm with an effective blade tip speed of 45.5 m/min. Slurry density was maintained at 60 to 70 pct solids. Attritioner overflow discharged onto a 250- μm vibrating screen. The oversize fraction was ground in a closed-circuit rodmill, and the undersize fraction was

classified in a 30-mm hydrocyclone. The cyclone overflow, about 75 pct minus 10 μ m, was discharged as a slime tailings, and the underflow, 95 pct plus 10 μ m and 85 pct of the feed, was discharged to a conditioner. H_2SiF_6 and a fatty acid-pine oil emulsion were added to depress phosphate minerals and to collect carbonate minerals for flotation. In the first flotation circuit, the float product was rejected as carbonate tailings and the underflow was fed with an amine acetate solution directly to the silica flotation cells. Silica tailings were floated, and the final underflow was the phosphate concentrate. Conditioning and flotation were carried out in 7-liter flotation cells.



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FIGURE 5 - Continuous-circuit beneficiation of partially altered phosphatic shale

Tests were made in this equipment on a 10-ton bulk sample of shale from the Area C stockpile (not the same sample as used in the bench-scale tests above). The shale contained 24.8 pct P_2O_5 , 36.9 pct CaO, 20.6 pct SiO_2 , 1.08 pct MgO, and 3.8 pct organic C. Average results from hourly samples taken during two 6-hour continuous tests are shown in table 5. In these tests, 71 pct P_2O_5 recovery was achieved at a concentrate grade of 30 pct P_2O_5 . Calcining to remove organics, as practiced in the western field, generally increases concentrate grade 2 to 3 pct. The MgO analysis (0.9 pct) is high compared with present levels (0.6 to 0.8 pct). However, future utilization will require handling higher MgO levels in fertilizer processing or subjecting concentrates to a pretreatment for selective removal of MgO (14).

TABLE 5 - Average results of two continuous-circuit tests on a bulk sample from the Area C stockpile following schematic flowsheet shown in figure 5

Product	Weight, percent	Metallurgical Data								CaO/P ₂ O ₅ ratio
		Analyses, percent				Distribution, percent				
		P ₂ O ₅	CaO	SiO ₂	MgO	P ₂ O ₅	CaO	SiO ₂	MgO	
Phosphate concentrate ^{1/}	57.9	29.7	42.8	12.8	0.90	71.3	69.5	33.8	49.6	1.44
Carbonate float	8.6	20.9	32.0	25.8	1.52	7.4	7.7	10.1	12.5	1.53
Silica float	18.6	17.6	26.7	36.3	1.22	13.6	13.9	30.8	21.6	1.52
Slimes	<u>14.9</u>	<u>12.5</u>	<u>21.3</u>	<u>37.3</u>	<u>1.15</u>	<u>7.7</u>	<u>8.9</u>	<u>25.3</u>	<u>16.3</u>	<u>1.70</u>
Composite/totals	100.0	24.1	35.7	21.9	1.05	100.0	100.0	100.0	100.0	1.48

Operating Conditions

Operation	Conditions		Reagents, kg/t ore		
	Time, minutes	pH	H ₂ SiF ₆	Fatty acid ^{2/}	Amine acetate ^{3/}
Condition	3.5	6.9	0.4	0.9	
Carbonate float	3.5				
Condition	.5	6.9			0.6
Silica float	4.5				

^{1/} 1.04 pct Al₂O₃, 0.60 pct Fe₂O₃.

^{2/} Emulsion: 200 g tall oil-fatty acid, 2.7 g pine oil, 2.7 g NaOH, 3.8 liters H₂O.

^{3/} Solution: 1 pct coco amine acetate, 1 pct tallow amine acetate.

SUMMARY AND CONCLUSIONS

A carbonate-silica flotation procedure for beneficiating low-grade western phosphate rock is being studied at the Bureau of Mines Albany Metallurgy Research Center. The procedure was applied to several different partially altered materials in bench-scale tests, and concentrates were produced that contained from 26 to 31 pct P₂O₅ with 57 to 85 pct P₂O₅ recovery. Concentrates grading 30 pct P₂O₅ with 71 pct P₂O₅ recovery were produced from a low-grade, partially altered shale in 30-kg/hr continuous-circuit tests.

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