CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

74-37 July 1974

BENEFICIATION OF PHOSPHATE ROCK FROM FERNIE, B.C.

(PROJECT MP-IM-6602)

by

F.H. Hartman and R.A. Wyman
Mineral Processing Division

Mines Branch

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MINES BRANCH INVESTIGATION REPORT

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Note: This report relates essentially to the samples as received. The report and any correspondence connected therewith shall not be used in full or in part as publicity or advertising matter.

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BENEFICIATION OF PHOSPHATE ROCK FROM FERNIE, B.C.

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F.H. Hartman* and R.A. Wyman**

SUMMARY

Work with a low-grade (10 to 11% P_2O_5) phosphate rock indicated maximum grades and recoveries of P_2O_5 were (1) by attrition 30.8% and 31.5%, (2) by flotation with soda ash, oleic acid and hot conditioning, 28.5 to 29.0% and 49%.

Results did not warrant a full scale investigation. National interest in a Canadian source of phosphate material suggests new techniques be tried with fresh sampling of this and other deposits.

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Work on rock from a phosphate prospect at Lodgepole Creek, near Fernie, B.C., was formally requested in May, 1966, by C. Warren Hunt, Staff Geologist, Western Co-Operative Fertilizers Limited, Calgary.

Two samples were received. The larger was taken from a deep bulldozer trench and the second was made up of core samples from three test holes. Both samples were low in phosphate content when compared with ore mined across the border in Idaho(1). A project was initiated in an attempt to economically beneficiate the material.

SAMPLES

1. Surface

The surface sample was taken from Location H 85 T. It consisted of 17 numbered, plastic-lined bags of material. Each bag represented an equal thickness of bed so that a range for evaluation purposes was achieved. The total thickness covered was 6.4 feet, thought to indicate recoverable material by surface mining. No significant changes in weathering or hardness had been noted through 10 vertical feet of the deposit which was under two feet of overburden.

Material, as received, was a dark brown colour, mostly earthy in texture with lumps up to 2-in size. A representative portion of each bag was taken and well blended. The composite was dried, screened on 28-mesh sieve and the oversize reduced in a hammermill, first with ½-in. and then with 1/8-in. grates. Heads assayed 10 to 11% P_2O_5 and contained in addition about 20% calcite, the remainder being quartz and feldspar with some mica and maffic minerals.

Black, rounded collophane particles, concentrated in the 65- and 100-mesh screen fractions, were identified. These appeared to be very fine apatite with a cementing agent. They were isolated by heavy liquid separation.

Minus 28-mesh head material was used in the experimental program.

2. Diamond drill core

Later, approximately 100 lb of core was received. This came from three test holes: H 221 Q2, H 93UQ7, and H 93 TQ3 and H 93 TQ4.

Core descriptions were sent for H 221 Q2 and H 93U Q7. Some chemical data was included. Overall grade was not high.

Material from these was not used in any tests for this project.

ANALYSIS

Total P was determined by chemical analysis and reported as P_2O_5. Some samples were separated into "sink and float" fractions using tetrabromoethane (specific gravity 2.96).

PROCEDURE AND RESULTS

Screening

The minus 28-mesh head sample was screened into various sized fractions down to minus 200-mesh. Results are given in Table 1.

TABLE 1

Size distribution, minus 28-mesh head sample

Screen test 1

Tyler screen fractions		Wt %	P_2O_5 per cent	
minus	plus		analysis	distribution
28	35	4.0	12.95	4.6
35	48	15.6	14.70	20.3
48	65	15.4	16.74	22.9
65	100	15.0	14.76	19.6
100	150	10.9	11.20	10.9
150	200	8.7	8.62	6.7
200	-	30.4	5.55	15.0
Head (calcd)		100.0	11.25	100.0
Head (analysis)		-	10.98	-

Magnetic separation

The minus 28-mesh head sample was passed once, at 25 amperes, through the Jones wet magnetic mineral separator equipped with salient pole plates. Results are given in Table 2.

TABLE 2

Magnetic fractionation, minus 28-mesh head sample

Jones test 1

Fraction	Wt %	P_2O_5 per cent	
		analysis	distribution
magnetics	1.2	7.73	0.9
middlings	28.6	10.70	27.8
non-magnetics	70.2	11.20	71.3
Head (calcd)	100.0	11.01	100.0

Attrition

Minus 28-mesh feed, at 33% pulp density, was ground in a Denver attrition cell at 1200 rpm for periods of 5, 10 and 15 minutes. The products were sized by screening and the results are shown in Table 3.

TABLE 3

Attrition tests 1, 2 and 3, minus 28-mesh head samples

Denver attrition cell, 33% solids, 1200 rpm, time varied

Test		1			2			3		
Time, minutes		5			10			15		
Tyler screen fractions		Wt %	P_2O_5 per cent		Wt %	P_2O_5 per cent		Wt %	P_2O_5 per cent	
minus	plus		anal	dist		anal	dist		anal	dist
28	48	11.2	18.62	18.0	9.9	19.26	16.8	9.4	19.55	16.0
48	65	10.8	19.79	18.6	10.4	20.43	18.6	10.3	21.18	19.0
65	100	13.1	19.76	22.5	12.2	19.84	21.2	11.9	21.23	22.0
100	150	10.0	14.12	12.1	9.5	15.07	12.5	9.2	15.14	12.1
150	200	8.3	9.94	7.2	7.6	10.55	7.0	7.6	10.87	7.2
200		46.6	5.36	21.6	50.4	5.41	23.9	51.6	5.28	23.7
Heads (calcd)		100.0	11.54	100.0	100.0	11.40	100.0	100.0	11.43	100.0

For test 4, minus 200-mesh material was screened from the sample. The plus 200-mesh was ground for 15 minutes at 1200 rpm in the Denver cell.

Test 5 was similar to test 4, except that plus 200-mesh material was stirred and treated for one hour in an ultrasonic bath and again screened before the plus 200-mesh fraction was attrition ground.

Results are given in Table 4.

TABLE 4

Attrition tests 4 and 5, screened head samples

15 minutes in Denver attrition cell, 33% (solids), 1200 rpm

Test		4			5		
Conditions		Screened on 200 mesh before* and after** attrition grinding			Screened on 200 mesh before* one hour ultrasonic treatment; before**, and after*** the following attrition grinding		
Results Tyler screen fractions		Wt %	P_2O_5 per cent		Wt %	P_2O_5 per cent	
minus	plus		anal	dist		anal	dist
28	48	8.2	20.77	15.0	3.1	29.27	8.0
48	65	9.6	22.30	18.8	4.6	30.84	12.8
65	100	11.5	20.66	20.9	6.8	30.76	18.8
100	150	8.8	16.38	12.7	5.0	27.31	12.4
150	200	7.3	10.95	7.0	5.0	19.18	8.5
200		40.9*	5.33	19.2	43.9*	5.21	20.5
200		-	-	-	29.1**	6.22	16.3
200		13.7**	5.31	6.4	2.5***	11.97	2.7
Heads (calcd)		100.0	11.37	100.0	100.0	11.14	100.0

The heads in test 6 were screened and the plus 200-mesh fraction was attrition ground in a Waring blender, at low speed, for two minutes at about 40% solids. Results are given in Table 5.

TABLE 5

Attrition test 6, screened head sample

2 minutes in Waring blender, 40% solids, low speed

Conditions		Screened on 200 mesh before* and after** attrition grinding		
Results Tyler screen fractions		Wt %	P_2O_5 per cent	
minus	plus		analysis	distribution
28	48	5.8	23.71	12.2
48	65	7.5	24.64	16.4
65	100	9.2	24.70	20.2
100	150	7.3	18.60	12.1
150	200	6.7	13.49	8.0
200*		39.7	5.51	19.5
200**		23.8	5.48	11.6
Head (calculated)		100.0	11.23	100.0

In tests 7 and 8, the plus 200-mesh screened fraction (about 3.6% solids) was stirred and treated in an ultrasonic bath for 1 hour. For test 8, the plus 200-mesh fraction (25% solids) was further treated with an ultrasonic probe for 10 minutes. Results are given in Table 6.

TABLE 6

Attrition tests 7 and 8, screened head samples

Ultrasonic treatment

Test		7			8		
Conditions		Screened on 200 mesh before* and after** ultrasonic treatment			Screened on 200 mesh before* and after** ultrasonic bath, and after*** ultrasonic probe treatment		
Results Tyler screen fractions		Wt %	P_2O_5 per cent		Wt %	P_2O_5 per cent	
minus	plus		anal	dist		anal	dist
28	48	3.2	29.22	8.3	3.0	29.30	8.0
48	65	5.0	30.95	13.6	4.7	31.11	13.2
65	100	6.6	30.86	18.1	6.9	30.53	18.3
100	150	5.3	26.67	12.5	4.9	26.89	11.9
150	200	5.2	18.64	8.6	4.9	19.41	8.6
200		41.3*	5.42	19.8	42.4*	5.35	20.4
200		33.4**	6.45	19.1	30.5**	6.06	16.6
200		-	-	-	2.7***	12.47	3.0
Heads (calcd)		100.0	11.29	100.0	100.0	11.13	100.0

Ultrasonic treatment

A 500-gram head sample was wet screened on a 325-mesh screen and the plus 325-mesh fraction treated by stirring in an ultrasonic bath. After varying time intervals, starting with 10 minutes, the sample was screened into minus 325-mesh "cuts" of freed material and plus 325-mesh fractions that were returned for further attrition. As treatment continued, time periods were increased until 4 hours was used for cuts 21 to 24.

Screen analyses of plus 325-mesh fractions were run after the 14th and 24th cuts; screen products from the former were combined for further comminution.

Screen products from the 24th cut were combined and separated using heavy liquids (2.96 specific gravity). Some unexplained weight loss which occurred during this treatment is presumed to be due to material that dissolved in the heavy liquids (tetrabromoethane and carbon tetrachloride). The sink fraction was examined mineralogically and selected size particles analyzed chemically.

Other selected cuts were analyzed for P_2O_5.

Results are given in Tables 7 and 7A.

TABLE 7

Ultrasonic comminution, screened head sample. Ultrasonic test 1

Plus 325-mesh feed, fines removed

Cut	Time min	Minus 325-mesh		
		Wt %	P_2O_5	Remarks
1	-	28.5	5.04	Screened before ultrasonics
2	10	20.1	4.41	
3	10	5.2	3.86	
4	10	3.5	5.71	
5	10	2.6	6.40	
6	10	1.7	-	
7	10	1.2	-	
8	20	1.7	-	
9	20	1.6	-	
10	20	1.2	9.80	
11	30	1.5	-	
12	30	1.2	-	
13	60	1.3	-	
14	60	1.2	13.09	plus 325-mesh screened
15	60	0.6	-	
16	120	1.0	-	
17	120	0.7	-	
18	120	0.6	-	
19	120	<0.1	-	blank-stirring, no ultrasonics
20	180	0.9	-	
21	240	1.4	-	
22	240	1.4	20.99	"oil" noticed
23	240	1.4	-	
24	240	0.8	-	plus 325-mesh screened
Sub total	1980	81.4	-	
ungground plus 325-mesh				
Float 2.96	-	8.0	19.09	
Sink 2.96	-	10.6	34.81	
Sub total		18.6	28.04	
Total		100.0	-	

TABLE 7A

Size distribution, plus 325-mesh fraction

Cuts 14 and 24, from Table 7

Cut		14	24*
Tyler screen fractions		Weight %	Weight %
minus	plus		
-	35	1.9	1.2
35	48	6.7	7.0
48	65	15.6	17.1
65	100	20.9	24.7
100	200	28.5	30.8
200	-	26.4	19.2
Total		100.0	100.0

Note: * rounded, sized black ovules, free quartz, few quartz grains surrounded by black.

Sink fraction (sp gr 2.96)

- clear white particles - apatite + pyroxene
- 35 + 65 mesh black pellets: P_2O_5 - 35.80%, total Ca - 35.65%
  LOI - 4.64%, C Cl_4 soluble - 0.66%

Superpanner

A series of cuts from the heads were made using a superpanner.

Results are shown in Table 8.

TABLE 8

Superpanner fractionation, head sample. Superpanner test 13.

Cut	Wt %	P_2O_5 per cent		Remarks
		analysis	distribution	
1	8.8	4.83	3.6	slimes
2	4.0	4.57	1.6	fines
3	4.6	4.43	1.7	brown-earthy
4	13.1	4.67	5.3	brown-earthy
5	6.7	5.73	3.3	brown
6	57.0	14.88	72.9	brown, black, white
7	5.8	23.34	11.6	brown, black, white
Total	100.0	11.65	100.0	

Flotation

Conditions for the tests run were:

- solids, 20-25% in a 500-gram Denver Sub-A laboratory flotation cell
- grinding, using 500-gram samples at 50% solids in a medium size (8.75 x 9.60-in.) Abbé mill, at 80 rpm.
- media, consisted of 3000 grams ½-in. burundum (fused alumina) "cylpebs".

An attempt was made to float unground minus 28-mesh heads in a pulp made alkaline with soda ash and sodium silicate, using an emulsion mix for collector (6:6:1:: fuel oil: distilled oleic: cyanamid 801). No recovery was observed.

Ground ore, floated with soda ash and oleic acid required large quantities of collector and left high rougher tailings. Finer grinds lowered the P_2O_5 distribution in the tailings.

Hot conditioning of ground ore with soda ash and distilled oleic acid was tried. The pulp, with reagents added was heated to boiling temperatures before flotation. Collector consumption remained high but better grade concentrates were made and loss of P_2O_5 in the tailings was less.

In test 7, the head sample was ground for 30 minutes, then screened on a 325-mesh sieve. The plus 325-mesh fraction was hot conditioned with soda ash (8 lbs per ton) and distilled oleic acid (12 lbs per ton). Flotation produced a heavy, voluminous froth that was cleaned twice.

In test 8, the head sample was treated for 1 hour in an ultrasonic bath with stirring, then screened on a 325-mesh sieve. The plus 325-mesh fraction was ground for 30 minutes and a procedure similar to test 7 repeated.

Results are shown in Table 9.

TABLE 9

Flotation tests 7 and 8

30 minute grind, hot conditioning

Test	7			8		
Conditions						
Reagents,	lbs per ton			lbs per ton		
Soda ash	8			8		
Oleic acid	12			12		
Remarks	Ground, screened and plus 325-mesh fraction hot-conditioned.			ultrasonic treatment, screened, plus 325-mesh fraction ground and hot-conditioned.		
Results	Wt %	P_2O_5 per cent		Wt %	P_2O_5 per cent	
Fraction		analysis	distribution		analysis	distribution
-325 mesh (slimes)	75.3	8.61	58.9	60.6	4.95	28.0
Concentrate	11.1	23.88	24.1	10.1	28.73	27.2
Cleaner 2 tailings	2.6	16.07	3.7	7.3	25.19	17.2
Cleaner 1 tailings	2.6	12.34	3.0	10.6	17.77	17.6
Rougher tailings	8.4	13.39	10.3	11.4	9.39	10.0
Head (calculated)	100.0	11.00	100.0	100.0	10.70	100.0

Test 8 was repeated but without ultrasonic treatment; also the additional fines produced during grinding were removed before hot conditioning. Results using this procedure were not as good as those in the previous test. X-ray diffraction analysis of the concentrate indicated it was composed of apatite with minor amounts of quartz and calcite. (Mineral Processing Division Mineralogical Report, Sample MP-MIN-963).

A 45 minute grind of plus 325-mesh material, using 8 lb per ton soda ash and 12 lb per ton distilled oleic acid with hot conditioning followed by an additional 8 lb per ton soda ash to cleaner 1, gave lower tailings and a concentrate assaying 27% P_2O_5. A 15 minute grind of plus 325-mesh material, then removing the minus 325-mesh product, followed by a further 45 minute grind and hot conditioning gave similar grades of rougher tailings and concentrate.

In test 21, the head sample was screened on a 200-mesh sieve. The plus 200-mesh fraction was ground for 45 minutes and hot conditioned with 8 lb per ton soda ash and 12 lb per ton distilled oleic acid. After the rougher float, 8 lb per ton soda ash and 3 lb per ton distilled oleic were added to cleaner 1. The concentrate was cleaned 3 times. Test 22 was a repeat of test 21, except that 6 lb per ton oleic acid were added to the first cleaner. Results are compared in Table 10.

TABLE 10

Flotation tests 21 and 22

200-mesh screens, plus fraction ground 45 minutes; hot conditioned

Test	21			22		
Conditions						
Reagents	lb per ton			lb per ton		
Soda ash-rougher	8			8		
-cleaner 1	8			8		
Oleic acid-rougher	12			12		
-cleaner 1	3			6		
Remarks	Hot conditioning in rougher float only					
Results	Wt %	P_2O_5 per cent		Wt %	P_2O_5 per cent	
Fraction		analysis	distribution		analysis	distribution
minus 200 mesh	38.3	5.51	19.2	39.1	5.39	19.2
Concentrate	3.5	29.01	9.3	7.1	25.74	16.7
Cleaner 3 tailings	2.1	29.94	5.8	3.9	25.72	9.2
Cleaner 2 tailings	5.7	27.87	14.5	6.3	25.31	14.6
Cleaner 1 tailings	12.3	17.70	19.7	5.1	16.94	7.9
Rougher tailings	38.1	9.05	31.5	38.5	9.25	32.4
Heads (calculated)	100.0	10.97	100.0	100.0	10.96	100.0

Replacing soda ash with sodium silicate gave better froth control. The reagent Lomar D added after hot conditioning with soda ash and oleic acid changed froth conditions; from results its use did not appear economical.

In test 28, the screened 200-mesh plus fraction was treated ultrasonically while stirring for one hour, screened again on a 200-mesh sieve and the plus material ground for 45 minutes. The pulp was then treated in a similar manner to that in test 21 (Table 10).

Test 29 was similar to test 28 except that a 150-mesh sieve replaced the 200-mesh screen. The combined minus 150-mesh fractions were screened on finer mesh screen sizes and analyzed.

Results of test 28 and test 29 are shown in Table 11.

TABLE 11

Flotation tests 28 and 29

Screened 200 or 150 mesh, treated ultrasonically, plus fractions ground 45 minutes, hot conditioned.

Test	28			29		
Conditions						
Reagents,	lbs per ton			lbs per ton		
Soda ash-rougher	8			8		
-cleaner 1	8			8		
Oleic acid-rougher	12			12		
-cleaner 1	3			3		
Remarks	Screened 200 mesh* ultrasonic treatment, screened 200 mesh **, ground 45 min, hot conditioned.			Screened 150 mesh* ultrasonic treatment, screened 150 mesh** ground 45 min, hot conditioned.		
Results	Wt %	P_2O_5 per cent		Wt %	P_2O_5 per cent	
Fraction		analysis	distribution		analysis	distribution
minus 150 mesh	-	-	-	44.0*	6.10	24.4
" 150 mesh	-	-	-	29.2**	6.23	16.5
minus 200 mesh	41.8*	5.14	19.8	-	-	-
" 200 mesh	24.9**	5.41	12.4	-	-	-
Concentrate	13.0	29.38	35.3	10.3	29.36	27.5
Cleaner 3 tailings	4.7	27.14	11.8	4.2	29.84	11.3
Cleaner 2 tailings	3.7	22.92	7.9	4.3	27.12	10.7
Cleaner 1 tailings	3.8	13.23	4.6	3.8	16.39	5.6
Rougher tailings	8.1	10.99	8.2	4.2	10.41	4.0
Heads (calculated)	100.0	10.84	100.0	100.0	11.01	100.0

*plus **

Combined minus 150 mesh fractions			
-150 +200	10.6	11.10	19.1
-200 +325	21.0	7.28	24.9
-325	68.4	5.03	56.0
Total	100.0	6.14	100.0

Other collectors tried were Igepon TE-42, and Armour RD 3312/Neo Fat 94-04 combination. Neither gave results that were as good as those using the oleic acid/soda ash - hot conditioning combination. In one test rougher tails were low, using the Armour products.

DISCUSSION

As work progressed it became apparent the 10-11% P_2O_5 sample submitted was low grade, ".... sample with a P_2O_5 content of 9.2 per cent is extremely low grade, and it might be advisable to consider wasting this material."(1). The following information (2) is indicative of Idaho ore grades that have been classified as "High grade, (>31 pct P_2O_5), Intermediate grade, (24 - 31 pct P_2O_5), Marginal grade (18 - 24 pct P_2O_5), Low grade (<18 pct P_2O_5)."

Although this project was not carried to the ultimate conclusion, a number of interesting aspects warrant comment.

The highest grade product was obtained using a mechanical treatment, attrition and ultrasonics. This occurred in the 65- and 100-mesh fractions. Results for three tests are summarized in Table 12.

TABLE 12

Grade and recovery using mechanical treatment-attrition and ultrasonics.

Screened feed

Table		4		6		6	
Test		5		7		8	
Tyler screen fractions		P_2O_5 per cent		P_2O_5 per cent		P_2O_5 per cent	
minus	plus	analysis	distribution	analysis	distribution	analysis	distribution
48	65	30.84	12.8	30.95	13.6	31.11	13.2
65	100	30.76	18.8	30.86	18.1	30.53	18.3
48	100	30.79	31.6	30.89	31.7	30.76	31.5

The similarity of these results suggests that the maximum recovery possible by attrition is 31.5% at 30.8% P_2O_5.

Intensive ultrasonic treatment (Table 7) gave a final product of 28.04% P_2O_5, which on treatment with heavy liquid gave a sink fraction assaying 34.81% P_2O_5. Black particles selected from the sinks (Table 7A) analyzed as follows:

P_2O_5	-35.80%
Ca (total)	-35.65%
LOI	- 4.64%
C Cl_4(soluble)	- 0.66%

If the calcium is considered to be present as apatite and $CaCO_3$, the loss on ignition calculated from $CaCO_3$ is 3.90%. This amount added to the loss on ignition from organics, 0.66% (CCl_4 soluble content), amounts to 4.56% (4.64% by analysis).

Flotation results using hot conditioning with soda ash and oleic acid show the importance of screening and grinding the feed to free and clean surfaces. Results are even better when ultrasonics are included. Table 13 show the results of three tests.

TABLE 13

Grade and recovery by flotation

Screened feed, 45 minute grind, with and without ultrasonics

Table	10		11		11	
Test	21		28		29	
Ultrasonics	no		yes		yes	
	P_2O_5 per cent		P_2O_5 per cent		P_2O_5 per cent	
Fraction	analysis	dist	analysis	dist	analysis	dist
Concentrate	29.01	9.3	29.38	35.3	29.36	27.5
Cleaner 3 tailings	29.94	5.8	27.14	11.8	29.84	11.3
Cleaner 2 tailings	27.87	14.5	-	-	27.12	10.7
Composite	28.60	29.6	28.78	47.1	28.95	49.5

Flotation testing was not carried further. Roasting of the samples to 500 and 700°C, before flotation, would have removed organic material (2). It is an expensive procedure and was not thought to be economical.

The approach to beneficiation using hot conditioning, although a costly step, appears promising. Had head grade been higher it could possibly eliminate some of the extended cleaner steps required with conventional tall oil or oleic acid floats. It could possibly also eliminate the acid treatment to kill the froth when an amine float is required to remove silica with certain phosphate ores. In general, hot conditioning is more selective, which should also apply when quartz is present.

The results suggest that concentration by attrition and/or other mechanical means, followed by heavy-liquid separation should be considered. In view of current national interest in a Canadian source of phosphate material this and other low-grade ores should be re-evaluated using fresh techniques.

CONCLUSIONS

An investigation, not carried to completion because of the low-grade head sample (10 to 11% P_2O_5), showed maximum recovery and grade figures to be:

(1) By mechanical treatment - 31.5% and 30.8% P_2O_5

(2) By flotation - 49% and 28.5 - 29.0% P_2O_5.

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