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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

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Mines Branch Investigation Report IR 58-157

BENEFICIATION INVESTIGATION OF PYROPHYLLITE BEARING ROCKS FROM MANUELS, NEWFOUNDLAND

by

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IR 58-157

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Summary

Beneficiation tests were conducted on two samples of pyrophyllite-bearing rock from Newfoundland. One sample, composed chiefly of muscovite and pyrophyllite cannot be easily beneficiated to the specifications set; the other can readily be improved to a good quality product. It is possible that by partial beneficiation of the first followed by blending with some of the second, a product would be obtained very close to that desired.

Introduction

Four samples were submitted (by Mr.Gover,Deputy Minister, Newfoundland Department of Mines and Resources). These were described by Mr.Gover as follows: -

"Sample No.1 Oval, is a barrel containing about 450 lb. of crude, primary crushed pyrophyllite having a low alumina (10-16%) and low alkali (less than 1%) content".

"Sample No.2 Mine Hill, is a barrel containing about 450 lb. of crude, primary crushed pyrophyllite having a high alumina (over 20%) and a high alkali (over 1%) content".

"Sample No.3 Milled Product, is a 100 pound bag of pyrophyllite as produced in the mill from high grade raw material (over 20% alumina and less than 1% alkali).

"Sample No.4 Country Rock is a 50 lb. bag of pyrophyllitized conglomerate which will have to be thrown out as waste by hand sorting".

The following partial chemical analysis by Mr. Ewart C.Bushe of Newfoundland Minerals Limited was submitted:

Sample No.	% Al_2O_3	% L.O.I.	% Total Alkalies
1. Oval	15.47	3.02	0.42
2. Jonnies Quarry (Mine Hill)	31.74	5.16	4.00
3. Production	21.47	4.11	0.48

Mineralogical Examination

This is described in detail in Mines Branch Investigation Report IR 58-109 by R.M.Buchanan.

Sample No.1, Oval

The sample is composed of irregular fragments of greyish-white material with maximum size of 2-3 inches. Uniform in appearance. Quartz, pyrophyllite, kaolinite and muscovite were identified. Most of the pyrophyllite flakes are smaller than 0.02 mm.

Sample No.2, Mine Hill

This sample is made up of irregular, elongated yellowish-green fragments with a splintery fracture. It is composed chiefly of a microgranular aggregate of muscovite and pyrophyllite with a small amount of quartz.

Sample No.3. Milled Products

This sample was received in the form of a very fine white powder. Pyrophyllite is the chief constituent, accompanied by quartz and a small but detectable amount of muscovite.

Chemical Analysis

Chemical analyses were carried out in order that the mineralogical compositions could be calculated.

Chemical Analysis (Mines Branch)

	Oval	Mine Hill	Milled Products
SiO_2	80.82%	56.01%	73.02%
Al_2O_3	14.38	31.70	21.22
K_2O	0.16	5.33	0.26
Na_2O	0.06	0.25	0.10
L.O.I.	3.01	4.80	3.99
Total	98.43	98.09	98.59
Weight Loss (Thermal Balance)	2.80	4.66	3.91

The determination of the weight loss by means of the thermal balance resulted in values slightly lower than those determined by the standard L.O.I. technique. The difference is principally due to the correction for the small amount of water taken up by the sample during handling.

The mineralogical composition of each sample was calculated in the following manner. The alkalies were first assigned to muscovite (potassium mica) and to paragonite (the sodium analogue of muscovite). After subtracting the requisite amounts of silica, alumina, and water (weight loss determination), the remaining alumina and water was proportionately divided between pyrophyllite (5.00% H_2O) and kaolinite (14.0% H_2O)

Calculated Mineralogical Compositions

Oval

	SiO_2	Al_2O_3	H_2O	K_2O	Na_2O	Total
Muscovite	0.62%	0.52%	0.06	0.16	-	1.36
Paragonite	0.30	0.35	0.03	-	0.06	0.74
Pyrophyllite	27.70	11.74	2.08	-	-	41.52
Kaolinite	2.10	1.77	0.63	-	-	4.50
Quartz(diff.)	50.10	-		-	-	50.10
Total	80.82	14.38	2.80	0.16	0.06	98.22

Mine Hill

	SiO_2	Al_2O_3	H_2O	K_2O	Na_2O	Total
Muscovite	20.6	17.3	2.04	5.33	-	45.3
Paragonite	1.5	1.2	0.15	-	0.25	3.10
Pyrophyllite	29.3	12.4	2.19	-	-	43.9
Kaolinite	0.9	0.79	0.28	-	-	2.0
Quartz	3.7	-	-	-	-	3.7
Total	56.0	31.7	4.66	5.33	0.25	98.0

Milled Product

	SiO_2	Al_2O_3	H_2O	K_2O	Na_2O	Total
Muscovite	1.01	0.85	0.10	0.26	-	2.22
Paragonite	0.59	0.49	0.06	-	0.10	1.24
Pyrophyllite	43.8	18.58	3.28	-	-	65.66
Kaolinite	1.54	1.30	0.47	-	-	3.31
Quartz(diff)	26.08	-	-	-	-	26.08
Total	73.02	21.22	3.91	0.26	0.10	98.51

Beneficiation

Investigation on beneficiation was carried out on the Mine Hill and Oval samples in order to obtain a product containing over 20% alumina and less than 1% alkali.

Flotation tests were done in the Denver laboratory cells.

With the Oval sample L.O.I's and X-ray diffraction patterns were used as a guide to the grade of flotation products. Chemical analyses were carried out on the most promising. In the Mine Hill material, X-ray diffraction patterns only were used as a guide to the comparitive separation of pyrophyllite from muscovite. This is because muscovite also has an L.O.I. The flotation data is shown in tabulated form.

Beneficiation of Mine Hill Material

The sample was crushed in a jaw crusher and ground in a ball mill, dry, to give the following screen analysis.

Mesh	%
+ 65	1.3
- 65 + 100	20.1
-100 + 150	19.5
-150 + 200	12.1
- 200	47.0

The above material was used in Flotations Nos. 1 - 10. To calculate the actual pyrophyllite recovery, complete chemical analysis is required as in the head sample.

TABLE I - MINE HILL

Flotation #	Product	% Wt. Dist.	X-Ray Order	K_2O	Na_2O	Al_2O_3	Reagents lbs/ton
1	Concentrate Tailings #1 Cleaner Tailings #2 Cleaner Tailings	32.7 31.8 32.2 3.3	2	2.22 4.68			0.42 Pine Oil 1.5 Sod.Silicate
2	Concentrate Tailings #1 Cleaner Tailings #2 Cleaner Tailings	20.2 38.4 29.0 12.4	1	1.60 4.60			0.37 Pine Oil 0.67 Sod.Silicate
3	Concentrate Tailings	65.5 34.5	4				0.37 Pine Oil 2.7 Sod.Silicate
4	Concentrate Tailings	55.2 44.8	5				0.37 Pine Oil 1.3 Sod.Silicate
5	Concentrate Tailing	58.7 41.3	3	2.78 4.77			0.37 Pine Oil 1.3 Sod.Silicate pH 5.9
6	Concentrate Tailing	55.0 45.0	6				1.3 Sod.Silicate 3.3 Dupanol
7	Concentrate Tailing	68.3 31.7	8				2.6 Sod.Silicate 6.6 Dupanol
8	Concentrate Tailings	56.9 43.1	7				1.3 Sod.Silicate 6.6 Dupanol
9	Concentrate Tailings Cleaner 1 Tailings	22.2 66.1 11.7		1.76	0.11	29.12	1.3 Sod.Silicate 0.8 220B
10	Concentrate Tailings Cleaner 1 Tailings	23.9 61.4 14.7		1.79	0.11	29.26	1.3 Sod.Silicate 0.8 220B

Table #1 contains the reagents used and the analytical results. Flotation #2 shows that reduction in the amount of Pine Oil used gives a better product but lower recovery than in #1.

Flotation Nos. 3 and 4 shows little separation.

Flotation #5 gives an indication of fairly good separation at pH 5.9 with no cleaning stage.

Flotation Nos. 6, 7 and 8 using Dupanol are not encouraging. It was noted that in #8 there was a slight pyrophyllite concentration in the tailings.

In Nos. 9 and 10, 220H was used. Although the total alkalis are slightly higher than in #2, the recovery is better and only one cleaning stage is involved.

In #10, when the necessary Al_2O_3 has been allocated to satisfy the K_2O in muscovite and Na_2O in pavagonite, and a nominal amount of 1% Al_2O_3 allowed for kaolinite, then % pyrophyllite in the concentrate = 79.5%. This gives a pyrophyllite recovery of 40.3%.

X-ray patterns on flotation products from fine grinds indicate separations that are almost as good but no better than #10 but with a greatly improved recovery. This was on material which was only 0.6% + 325 mesh.

Conclusions

1. The prospect of reducing the total alkalis (Na_2O + K_2O) to less than 1% and achieve a resonable recovery is not very encouraging.

2. The best result obtained was in flotation #10.

Al_2O_3	Total Alkalies	Pyrophyllite	Pyrophyllite Recovery
29.26	1.90	79.5	40.3

In the calculation of the above figures, 1% Al_2O_3 was allowed for the kaolinite content.

3. Indications are that a better recovery can be obtained on a finer grind. This may warrant further investigation.

4. Further work on lower pH values may be useful.

Beneficiation of Oval Material

The sample was crushed in a jaw crusher and then ground dry in a pebble mill.

In Flotations Nos.11, 12 and 13 the material was ground to -65 mesh.

In Flotations Nos.14, 15 and 16, the grind was taken to 90% -325 mesh. In Flotation #17, -48 mesh material was used.

For Nos. 18 and 19 the material was ground to -65 mesh to give the following analysis

Mesh	%
+100	4.1
-100 +150	11.3
-150 +200	11.8
-200+250	7.7
-250 +325	28.4
-325	36.7

Table #2 contains the reagents used and the analytical results.

Results on the Mine Hill material showed success with reagent 220B., which is used extensively in the investigation of the Oval sample.

In Flotation #11 grade material was produced but recovery was poor.

In Flotation Nos. 12, 13 and 15, an acid circuit was used with considerable success in #12.

In Flotation #16, fine material was used, -325 mesh. Grade material was produced with a recovery of 64.5%.

In Flotations Nos.18 and 19, the procedure on each was identical. The recoveries were low although grade material has been produced. This shows that a finer grind is necessary to improve recovery.

The alkali content in the products assayed has been reduced to less than 0.15%.

TABLE II OVAL

Flotation #	Product	L.O.I.	Al_2O_3	% Recovery	Na_2O	K_2O	Reagents lb/ton
11	Concentrate	4.18		44.4			220 R.0.4 lb/ton Sodium Silicate 1.3 lb/ton -65 mesh
	Tailings	2.75		32.2			
	Scavenger Concentrate	3.59		23.4			
12	Concentrate	4.10		53.6			220 R. 0.8 lb/ton pH 3.2 (HCl)-65 mesh
	Tailings	2.85		46.4			
13	Concentrate	3.39		92.2			Pine Oil 0.75 lb/ton pH 6.8 (H.F.) -65 mesh
	Tailings	2.76		7.8			
14	Concentrate	3.72		60.9			220 R. 0.8 lb/ton Sodium Silicate 1.3 lb/ton -325 mesh
	Tailings	2.46		11.2			
	Scavenger Conc.	2.96		27.9			
15	Concentrate	3.54		87.0			220 R. 0.8 lb/ton pH 1.2 (HCl) -325 mesh
	Tailings	2.13		13.0			
16	Concentrate	3.99	20.43	64.5			220 R. 0.8 lb/ton Sodium Silicate 1.3 lb/ton -325 mesh
	Tailings	2.48	11.39	19.3			
	Cleaner 1 Tailings	3.08	14.89	16.2			
17	Concentrate	4.08	20.57	33.0	0.05	0.09	220 R. 0.8 lb/ton Sodium Sil.1.3 lb/t -48 mesh
	Tailings	2.42	12.26	60.4			
	Cleaner 1 Tailings	3.42	16.21	6.6			
18	Concentrate	4.02	20.13	48.7	0.06	0.09	220 R. 0.8 lb/ton Sodium Silicate 1.3 lb/ton -65 mesh
	Tailings	2.34	11.24	41.2			
	Cleaner 1 Tailings	3.21	15.3	10.1			
19	Conc.	4.08	20.77	44.9	0.05	0.09	220 R. 0.8 lb/ton Sod.Sil.1.3 lb/ton -65 mesh
	Tailings	2.45	11.38	43.5	0.05	0.19	
	Cleaner 1 Tailings	3.16	15.78	11.6			

Samples of the Concentrate, Tailings and Heads of Flotation #16 were submitted for semi-quantitative spectrographic analysis with the following results -

	Tailings	Concentrate	Heads
Si	PC	PC	PC
Al	9	10	10
Fe	0.6	0.3	0.4
Ba	0.8	0.04	0.4
Ca	0.4	0.04	0.4
Ti	0.1	0.08	0.1
As(?)	0.2	0.2	0.2
Zr	0.06	0.01	0.02
Mg	0.1	0.06	0.06
Mn	0.03	0.007	0.02
Cu	0.05	0.008	0.01
Ni	0.06	0.005	0.009
Co	0.01	N.D.	N.D.

? - Identification not positive. These results show that all the trace elements have been concentrated in the tailings.

<u>Mixing</u>

As the Oval material is very low in alkali content, then by mixing with Mine Hill, until the upper limit of alkali content is reached, a considerable quantity of the Mine Hill material could be used.

If the Mine Hill concentrate was mixed with the Oval heads in equal parts a product containing 21.82% Al_2O_3 and 1.01% total alkali would be obtained. This may be a more profitable course to follow if the firing characteristics of the resulting product are satisfactory. i.e. Beneficiate only the Mine Hill material sufficiently, to mix with the Oval to give an acceptable product.

Conclusions

1. Grade material can be produced fairly easily. To obtain a reasonable recovery, fine flotation feed will be required, i.e. in the order of 325 mesh.

The best overall results was in Flotation #16 with a product assaying 20.43% Al_2O_3 and 64.5% recovery.

2. The quantity of alkali present in the concentrate products is very small, less than 0.15%.

3. Good results may also be obtained using an acid circuit.

4. Mixing the raw Oval material with a beneficiated Mine Hill product would likely be profitable.

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