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BENEFICIATION OF PYROPHYLLITE FROM NEWFOUNDLAND SUBMITTED BY THE AMERICAN ENCAUSTIC TILING CO., INC.

by

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Progress on this investigation was reported July 2, 1957 as I. M. Report No. 472. Since that time a considerable body of additional work has been done. About half the pyrophyllite may be recovered fairly easily by either flotation, or attrition with classification, at acceptable grades, but considerable difficulty is encountered in obtaining higher recoveries at acceptable grades.

Mineralogy

The sample consists essentially of pyrophyllite and quartz, the quartz being observed only in very fine particles. The coarser quartz is around 100 mesh but the bulk is much finer, generally of micron size.

Analyses

Although Al_2O_3 would normally be used as a standard L.O.I. is much more easily obtained, and for the purpose of this investigation the L.O.I. determination was used.

Object of Investigation

Pyrophyllite is theoretically composed of 28.3% Al_2O_3, 66.7% SiO_2 and 5.0% L.O.I. The feed used in the various tests ran from 3.0 to 3.2% L.O.I. which agreed substantially with the analyses provided by the American Encaustic Tiling Co., shown below:

SiO_2	78.6%
Al_2O_3	17.6%
L.O.I.	3.1%
Fe_2O_3	0.3%
Alkali	0.3%
	99.9%

The grade required for final product was 20% Al_2O_3, or about 71% pyrophyllite. Using L.O.I. a grade of 4.0% was sought, or 80% pyrophyllite, a good safety factor. With the feed averaging better than 60% pyrophyllite, the amount of upgrading required was not extensive and it was thought a recovery of 75% could be obtained without too much trouble. It was recognized, however, that quartz of the fineness being dealt with could be difficult to eliminate either by classification or by flotation.

Experimental Work

Since dry methods of working pyrophyllite are in common use, it was hoped that they could be successfully applied here. Initial results as reported in I.M. 472 indicated that screening, or air classifying, of the ground material would not be successful, but that careful light attrition with careful air classification was promising. The experimental campaign has since extended the dry attrition trials, has employed wet attrition and classification, and has tried flotation.

(1) Dry Attrition and Air Separation

(a) Rubber Balls (500 grams feed):

Feed for this test was obtained by crushing some of the pyrophyllite through 20 mesh and removing the fines so developed by air classification - 12% of the feed was classified off at a grade of 3.76% L.O.I. The coarse from air classification ran 2.93% L.O.I. and 500 grams of this was used for the attrition test.

The test consisted of 5 cycles. For each cycle the feed was ground 1 hour with 930 grams of rubber balls and the fines so

developed classified off in a Raymond Air Separator. Coarse from the air separator was returned to the mill for the next cycle. Results are shown below:

Fines from crushed feed	= 12% at 3.76% L.O.I.
Fines from attrition milling	= 33.6% at 4.37% L.O.I.
Coarse after attrition milling	= 54.4% at 2.39% L.O.I.
Recovery of pyrophyllite	= 59.6% at 4.21% L.O.I.

(b) Rubber Balls (1000 grams feed):

This test differed from (a) only in that 1000 grams of feed was used to begin the first grinding cycle, and 6 cycles were completed rather than 5, the grind for cycle 6 being three hours instead of one. Despite the longer attrition time, this test did not produce as much usable grade as (a).

Fines from crushed feed	= 12% at 3.76% L.O.I.
Fines from attrition milling	= 26.2% at 4.16% L.O.I.
Coarse after attrition milling	= 61.8% at 2.53% L.O.I.
Recovery of pyrophyllite	= 49.8% at 4.04% L.O.I.

(c) Rubber stoppers (2000 grams feed):

The attempt to speed up production by doubling feed rate as used in (b) did not work out. A new approach was therefore tried. For this a new lot of feed was crushed through 14 mesh and 2000 grams was placed in the mill together with 2000 grams of graded rubber stoppers. This charge was milled for 30 minutes, then removed and passed through the Federal Dust Classifier. Coarse from classification was returned to the mill for a second cycle, again with 2000 grams of graded rubber stoppers, and run for 1 hour. This test was stopped after the classification step for the second cycle because results were unsatisfactory.

Fines from attrition milling	= 32% at 3.88% L.O.I.
Coarse " " "	= 68% at 2.97% L.O.I.
Recovery of pyrophyllite	= 38% at 3.88% L.O.I.

(d) Rubber Stoppers (1000 grams feed):

A final dry attrition trial was made using an initial charge of 1000 grams - 14 mesh feed with 2000 grams of graded rubber stoppers. This test was carried through three cycles with grinds of 30 minutes, 1 hour, and 2 hours respectively. In each cycle fines were removed by Federal Dust Classifier. An improvement over test (c) was developed, but results were still not up to those for (a).

Fines from attrition milling	= 44.5% at 3.98% L.O.I.
Coarse " " "	= 55.5% at 2.80% L.O.I.
Recovery of pyrophyllite	= 53% at 3.98% L.O.I.

(2) Wet Attrition and Classification

In a preliminary trial of wet attrition, some feed crushed through 35 mesh was first classified in a teeter column, then the coarse from classification was ground in a pebble mill with a light charge of pebbles. Three such classification-grind cycles produced a product grading 3.87% L.O.I. for a 43% recovery.

(3) Dry and Wet Attrition Combinations

(a) A lot of the feed was prepared by crushing through 23 mesh. The -20 was air classified in the Raymond Classifier. Raymond coarse was then agitated with an impeller for 30 minutes, after which the fines were classified off. Two further cycles of 30-minute agitation and classification were completed before stopping the test.

Raymond fines	= 13.3% at 3.93% L.O.I.
Wet Classifier fines	= 36.4% at 3.96% L.O.I.
Coarse remaining	= 50.3% at 2.30% L.O.I.
Recovery of Pyrophyllite	= 62.8% at 3.94% L.O.I.

(b) A feed lot was prepared by crushing through 20 mesh. This was air classified in the Raymond Separator to remove fines. The

Raymond coarse was then put through five cycles of dry attrition milling with a charge of 930 grams of rubber balls with removal of fines by Raymond Separator following each milling. Raymond coarse remaining after the 5th cycle was pulped in a container and agitated for 30 minutes with an impeller, following which the fines were classified off. A second cycle had 30 minutes agitation, a third 60, a fourth 90, and a fifth 120 minutes; fines were classified off following each period of agitation. The final coarse product remaining after this treatment was comparatively low in L.O.I., 1.97%.

Raymond fines	= 12.0% at 3.76% L.O.I.
Dry attrition fines	= 33.6% at 4.37% L.O.I.
Wet attrition fines	= 9.3% at 4.53% L.O.I.
Final coarse	= 45.1% at 1.97% L.O.I.
Recovery of pyrophyllite	= 72.5% at 4.27% L.O.I.

(c) For the final test in this series, a feed lot was prepared by crushing through 20 mesh. This was air classified in the Raymond Separator to remove fines. The Raymond coarse was pulped with water and the fines classified off with no agitation. Following this came eleven cycles in which the coarse from each proceding step was pulped and agitated, then the fines classified off. Agitation times for these cycles were as follows: (1) 1 min., (2) 1 min., (3) 1 min., (4) 2 min., (5) 2 min., (6) 3 min., (7) 5 min., (8) 5 min., (9) 5 min., (10) 10 min., (11) 10 min. At this point the coarse was screened, wet, on 65 mesh and the -65 set aside as Tails No. 1. The +65 was passed through three more attrition cycles, each with 15 minutes agitation. Coarse was then again screened on 65 mesh with the -65 constituting Tails No. 2. The +65 was passed through one more attrition cycle, this time

with a 30 minute agitation time. The coarse remaining after classification constituted Tails No. 3.

Raymond fines	= 11.7% at 3.98% L.O.I.
Wet attrition fines	= 48.3% at 4.00% L.O.I.
Combined tails	= 40.0% at 1.78% L.O.I.
Recovery of Pyrophyllite	= 77.2% at 3.99% L.O.I.

(4) Flotation

(a) Minus 35 mesh feed:

Preliminary float trials were made using -35 mesh feed. Sodium silicate was used as a quartz depressant and two collectors were tried: Aeroamine 2026 and Nytron. In each trial the rougher froth was cleaned once. Recovery, as shown below, was poor, partly because of the coarse grind.

Collector	Recovery	Grade
Aeroamine 2026	33%	3.85% L.O.I.
Nytron	25%	4.00% L.O.I.

(b) Minus 100 mesh feed:

Two trials were next made using feed ground through 100 mesh and with Dupanol 80 as collector. Sodium silicate was again used to depress quartz. In the first trial Rougher froth was cleaned once using additional sodium silicate. In the second, two cleaning steps were used both with additional sodium silicate and Dupanol 80. Results were slightly better but still unsatisfactory.

Trial No.	Cleaners	Recovery	Grade
1	1	40%	4.00% L.O.I.
2	2	37%	3.99% L.O.I.

A combination test was also made in which feed ground through 20 mesh was first run through the Raymond Air Separator and the coarse from this was subjected to five cycles of dry attrition of 1 hour each with rubber balls, fines being air classified off

between each grind. Coarse after final air classification was wet ground through 100 mesh and floated with pine oil using HF. to depress quartz. This combination produced a fairly good result.

Raymond fines	= 12.0% at 3.76% L.O.I.
Dry attrition fines	= 33.6% at 4.37% L.O.I.
Flotation concentrate	= 12.0% at 3.57% L.O.I.
Tails	= 42.4% at 2.28% L.O.I.
Recovery of pyrophyllite	= 71% at 4.08% L.O.I.

(c) Minus 200 mesh feed:

A test with feed ground to -200 mesh was made using sodium silicate as quartz depressant and pine oil as collector. Step additions of the pine oil were used in Roughing and both sodium silicate and pine oil were added in each of the two cleaning steps. This test produced a somewhat higher recovery, but with lower grade.

	Recovery	Grade
Concentrates	54%	3.43% L.O.I.
Middlings	33%	2.98% L.O.I.
Tails	13%	2.04% L.O.I.

A somewhat more elaborate trial used -35 mesh feed to start. This was pulped and the fines classified off. Classifier sands were then wet ground to -200 mesh and floated with sodium silicate and Dupanol 80. The Rougher concentrate was passed through two cleaning steps each using additional sodium silicate and Dupanol 80. This treatment produced a lower grade tailing, but otherwise no overall improvement.

	Recovery	Grade
Classifier fines	24.6%	3.81% L.O.I.
Float Concentrate	22.5%	3.74% L.O.I.
" Middlings	32.2%	2.95% L.O.I.
" Tails	20.7%	1.99% L.O.I.

Recovery of pyrophyllite
(class. fines plus float conc.) = 47% at 3.78% L.O.I.

Comments

Throughout the investigation continuous difficulty was encountered in respect to the extremely fine grained nature of the quartz. Flotation techniques, usually successful on such materials were hampered because froths for all trials made were extremely voluminous (a characteristic of talcy materials). Despite quartz depressants, the fine quartz particles were continually swept up by mechanical entrapment in the froths, and could not be successfully dropped out in the cleaning stages. Flotation would probably best be applied to material from which part of the pyrophyllite had already been removed by dry working, or by dry working followed by some wet attrition. The advantage of flotation would be in speeding up the process time. One of the better results was obtained (see Flotation (b)) by following dry working with flotation (71% recovery at 4.08% L.O.I.). However, the process time for this trial was about 6 hours.

In contrast to this, the best overall result was obtained by following dry working with wet attrition (see Dry and Wet Attrition Combinations (c)). Here the process time was about 3 hours, half of which was in the latter stages where long attrition times were necessary and screening steps were introduced. If flotation were substituted for these latter stages, the process time would be materially reduced, and moderate grades could be accepted for blending with the high grade product obtained in the early stage of the wet attrition (classifier fines of up to 4.45% L.O.I. were produced).

Pyrophyllite's softness in comparison with quartz strongly suggests attrition as a logical approach. Here again the fine

size of the quartz interferes since either air or wet classification must be used to separate out the abraided material. Pyrophyllite is slightly heavier than quartz so that in classification only pyrophyllite particles smaller than neighbouring quartz particles can be successfully removed.

It was found that only by very slow working could satisfactory results be obtained. All attempts to speed up the attrition process ended in down-graded products.

Conclusions

A recovery of 77% of the pyrophyllite at a grade of 4.00% L.O.I. can be made by slow, multi-stage working of the material by wet attrition and classification. Dry working with air classification is successful if done slowly over long periods of time. Wet attrition followed by flotation appears to offer the most practical solution to this problem.

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