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INVESTIGATION OF CONCENTRATION OF GRAPHITE FROM MOUNT LAURIER, QUEBEC, SUBMITTED BY DR. L. J. La RUE

by

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INVESTIGATION OF CONCENTRATION OF GRAPHITE

FROM MOUNT LAURIER, QUEBEC

SUBMITTED BY DR. L. J. LaRUE

The sample received was in the form of lumps up to 6 inches in size, containing 22.7 per cent graphite. Information requested was for the production of a graphite concentrate.

Summary

This is a highly weathered material. Fresh material would probably require quite different treatment. With this sample, concentrates of approximately 97 per cent carbon may be made with a recovery of 65 per cent, or 90 per cent carbon concentrate with 92 per cent recovery, by simple processes.

Test Work

Investigations conducted were:

1. Preliminary mineralogical examination
2. Comminution methods
3. Concentration by flotation
4. General comments

1. Preliminary Mineralogical Examination

The material was friable and had been severely weathered. The following minerals were identified by microscopic and X-ray examination in approximate order of abundance.

Graphite

Quartz

Diopside

Goethite (+hematite?)

Sphene

Apatite

After crushing, a representative portion was passed through a micropulverizer. A high degree of liberation was achieved without reducing appreciably the size of the constituents.

The material was then screened and it was found that there was a concentration of graphite in the coarse sizes.

Screen Analysis

Mesh	Per cent
-8 + 28	4.2
-28 + 48	17.4
-48 + 65	12.7
-65 + 100	14.7
-100 + 200	24.5
-200	26.5

The quartz occurs as rounded, clear, colourless grains, and the diopside in the form of yellow stained cleavage fragments. The iron oxide is found to be in powdery fine-grained red masses. The sphene as flat irregular grains, and the apatite as clear colourless grains.

Absence of fresh material makes it difficult to speculate upon the nature of the deeper, unweathered portions of the deposit.

2. Comminution Methods

A preliminary result from the micro-pulverizer showed that the graphite flakes were concentrated in the coarser sizes. The comminution tests were performed with this objective, as the coarser the flake the greater the value. Also of importance is that the flake should be flat and clean, i.e., not be iron-stained or have attached gangue particles.

Tests were made using the Rolls, Victoria Mill, Hammer Mill, Micro-Pulverizer, Raymond Whizzer Pulverizer and Ball Mill.

A jaw-crusher was used to reduce the material to 1 to 2 inch lumps without appreciable damage to the flake. Products were examined under the microscope.

(a) Rolls

This gave a product containing a considerable quantity of particles where the gangue had been pressed into the graphite. This is undesirable in that it would be impossible to get a good liberation of gangue from graphite with this method.

(b) Victoria Mill

One pass through the Victoria Mill gave the screen analysis as shown; 1/8 inch grates were used and 16.5 per cent of the feed was lost as fines.

Mesh	Per Cent	Cumulative %
+14	2.2	2.2
-14 + 28	11.4	13.6
-28 + 65	38.4	52.0
-65 + 100	12.8	64.8
-100	35.2	100.0

Examination of the fractions showed that the graphite was not appreciably concentrated in the coarser sizes.

(c) Hammer Mill

The material was given one pass using 1/8 inch grates. A screen analysis was carried out and the fractions examined. There was a loss of 10 per cent due to fines.

Mesh	Per Cent	Cumulative %
+14	0.4	0.4
-14 + 28	4.7	5.1
-28 + 65	33.1	38.2
-65 + 100	16.0	54.2
-100	45.8	100

Although the screen analysis on this product approximates to that obtained on the micro-pulverizer, the concentrate of graphite in the coarser sizes is not good.

(d) Raymond Whizzer Pulverizer

(i) The setting of the Pulverizer was to give the coarsest product with all the hammers intact. Three products were obtained on the machine and a screen analysis made on each.

Mesh	Coarse	Medium	Fine
+ 14	-	-	-
- 14 + 28	0.5	0.4	-
- 28 + 65	18.4	4.4	1.9
- 65 +100	24.9	7.3	3.5
- 100	56.2	87.5	94.5

There was a good graphite concentration in the +100 mesh fractions. However, most of the sample was reduced to the 'Fine' state which was 94.5 per cent -100 mesh.

(ii) In the second test on the Raymond Whizzer all but two of the hammers were removed in an attempt to produce more in the coarse product. A screen analysis was made on each of the three products obtained.

Mesh	Coarse	Medium	Fine
+ 14	3.6	-	-
- 14 + 28	1.0	0.5	-
- 28 + 65	19.4	2.2	2.3
- 65 +100	25.1	14.4	3.8
-100	50.9	82.9	94.0

There was still less than 25 per cent of the total sample in the coarse product. It appears that the action of this machine is far too violent for this material.

A sample of the 'Fine' product was closely screened to -325 mesh.

Mesh	Per cent
+100	5.7
-100 + 150	9.3
-150 + 200	6.6
-200 + 250	5.7
-250 + 325	12.4
-325	60.3

In this material there was a good graphite concentrate down to +325 mesh. It seems that if the speed of this machine was reduced, results comparable to those obtained on the micro-pulveriser can be produced on a large scale.

The loss on this machine due to dust is even greater than with the Victoria or Hammer Mills.

(e) Ball Mill

Feed to Ball Mill 1 to 2 inch lumps 2250 gms.

Pebble charge - 7 lb

Solids - 40%

(1) The above charge was given a 10 minute grind and a screen analysis made. Only a little +8 mesh material remained.

Mesh	Per Cent	Cumulative %
- 8 + 28	5.7	5.7
-28 + 48	15.3	21.0
-48 + 65	14.3	35.3
-65 +100	13.9	49.2
-100 +200	20.7	69.9
-200	30.1	100.0

The +48 mesh material contains particles which have not reached the point of liberation which suggested that a longer grind was required.

(2) The material from the fine-grind was run for a further 10 minutes and screen analysis made.

Mesh	Per Cent	Cumulative %
-8 + 28	1.7	1.7
-28 + 48	8.0	9.7
-48 + 65	10.6	20.3
-65 + 100	14.8	35.1
-100 + 200	23.7	58.8
-200	41.2	100.0

The plus 200 mesh material showed a good graphite concentration with relatively clean flat flakes. There was no dust loss.

Conclusions on Comminution

It appears that the best overall results were obtained on the Ball Mill due to -

(a) During grinding the graphite remains as a coarse flake while the gangue is reduced in size. This effect was more marked with the Ball Mill than with the other types.

(b) The graphite flakes are much cleaner with Ball Milling than with the other type of equipment. It appears that the rubbing action detaches particles of gangue from the flakes and rubs off iron stains.

(c) The action of the pebbles produces a flat flake, whereas when the comminution was achieved by some means of impact, the flakes were twisted and curved.

(d) The problem of dust with ball milling is negligible compared with the other types of comminution.

3. Concentration by Flotation

The method of assay was by determining the Loss on Ignition (L.O.I.) by maintaining sample at 725°C for 24 hours. The L.O.I. was assumed to be the carbon content.

Flotation tests were carried out on products from the Rolls, Ball Mill and micro-pulveriser using Denver cells.

Flotation #1

The feed was a concentrated graphite product obtained by crushing through rolls and then air table treatment. Flotation and one cleaning operation produced a concentrate of 85.35 per cent carbon. Recovery in flotation was low, 35.9 per cent. Also recovery on the air table is low so the overall is very poor. Examination under the microscope revealed that the graphite flakes had adhering iron material and also gangue had been pressed into the flake. Thence continued cleaning by flotation would not be successful.

<u>Flotation #2</u> - -14 + 200 mesh.

The feed was obtained by giving 2 inch lumps a 20 minute grind in a ball mill. The product was screened on 14 and 200 mesh. There was very little material remaining on the +14 mesh. With one cleaning operation a concentrate of 91.21 per cent was obtained with 93.8 per cent recovery. A quantity of the flakes were iron stained which suggested for a very high grade product, further attrition would be necessary.

<u>Flotation #3</u> - -200 mesh

Feed in this test was the -200 mesh from the 20-minute ball mill grind. One cleaning gave a concentrate of 90.08 per cent and a recovery of 85.5 per cent.

Combining Flotations #2 and #3 an overall recovery was calculated.

<u>Ball Mill 20-Minute Grind</u>

Mesh	Screen Analysis	L.O.I. on Heads	L.O.I. on Concentrates	Recovery on Individual Fractions	Overall Recovery
-14 + 200	66.0	27.6	91.21	93.8	92.5
-200	34.0	13.3	90.08	85.5	

This shows that a good overall recovery, 92.5 per cent, and concentrate, over 90 per cent carbon, is made in one cleaning stage. Also shown is a concentration of graphite by ball milling in the -14 + 200 mesh heads.

Flotation #4 - -200 mesh.

This was to compare the effect of a shorter grinding time. Material was ground for 10 minutes. A concentrate of 83.26 per cent carbon with 87.9 per cent recovery was obtained with one cleaning stage. As expected this concentrate is not as clean as that obtained from the 20-minute grind, but recovery is slightly higher.

Flotation #5 - +200 mesh.

The feed was the same as Flotation #2. Reagents used were doubled in quantity. Five cleaning stages were used to study effect of repeated cleaning. NaOH was the only reagent added during the cleaning stages.

Only a slightly higher concentrate, 93.5 per cent carbon, was obtained with five cleaning stages, compared to 91.21 per cent with one cleaning stage. The recovery dropped considerably to 73.5 per cent. Microscopic examinations showed that most of the gangue present was as adhering particles to the graphite flakes and, therefore, further attrition would be necessary for a high grade product.

A screen analysis was made on the concentrate and an L.O.I. on each fraction.

Mesh	Per Cent Dist.	Per Cent L.O.I.
+ 65	46.0	94.42
- 65 + 100	20.1	92.15
-100 + 200	24.8	92.76
-200 + 325	7.3	93.68
-325	1.8	93.68

This shows a large proportion of the graphite is recovered in the larger sizes.

Flotation #6, 7 and 8

Feed was -14 + 200 mesh, 20 minute Ball Mill grind. After two cleaning stages the concentrate was reground for 10 minutes in the Ball Mill. It was refloated, cleaned once, given another 10-minute grind and floated again.

This procedure gave a concentrate of 97 per cent carbon with a recovery of 64.4 per cent. A screen analysis was done on the concentrate and an L.O.I. on each fraction.

Mesh	Per Cent Dist. of Graphite	L.O.I.
+ 48	20.0	97.2
- 48 + 100	42.2	97.2
-100 + 200	28.6	97.05
-200 + 325	6.9	97.10
-325	2.3	93.85

The above table shows that the gangue is differentially ground to a size finer than that of the bulk of the graphite. Over 60 per cent of the graphite is plus 100 mesh.

Flotation #9, 10 and 11

The feed in #9 was obtained by preliminary flotation in a large cell. Starting material was -14 mesh rolls product. No reagents were used in #9 flotation. The concentrate from #9 was divided and ground for 20 minutes in a Ball Mill at 70 per cent solids and 40 per cent solids, respectively, and refloated.

The lower pulp density grind gives a higher grade product with a lower recovery. These results also clearly show that when crushing is by use of the rolls, it is much more difficult to obtain a graphite concentrate than when initial grinding is by Ball Mill.

Flotation #12

Flotation of the micro-pulverizer product did not yield a good concentrate. There were a lot of flakes with adhering gangue, and the degree of differential breakdown between graphite and gangue appeared to be much less than with the ball mill.

Conclusions on Flotation

1. The highest grade and most easily won graphite was that in which comminution was effected by ball-milling.

2. Good results were obtained with the reagents used, which were Pine Oil, Kerosene and Caustic Soda. The addition of caustic soda does not have a very marked effect and may not be necessary.

3. Quite a large percentage of the graphite can be recovered in a coarse flake.

4. For purposes of calculation, recoveries were based on concentrate only. The cleaner tailings however contain considerable graphite which could be recovered if re-ground.

5. Due to difficulty of removing all concentrate from the cell, there was always an amount adhering to the sides which was washed into the tailings. In continuous running this loss will be greatly reduced.

General Comments

It has been shown that a high grade graphite concentrate can be obtained relatively easily.

After initial crushing to about 2 inch lumps, the best method of comminution was by ball-milling.

The required plant will depend upon the grade of concentrate required and whether an intermediate grade can be marketed.

If a 90 per cent grade is acceptable, this could be obtained with one ball-milling stage and one cleaning stage in the flotation circuit. If a higher grade is required, then provision must be made for regrinding.

The flotation tests were done on material which had been screened on 200 mesh. This screening could probably be replaced by use of a desliming unit.

Should a lower grade product, say 80 per cent, be marketable along with a high grade, a high recovery can be achieved.

The advantage of ball-milling on the other types are:-

1. Rubbing action of the balls clean adhering gangue from the flakes.
2. By the same action, the graphite flakes are flattened.
3. Dust problem reduced.
4. Differential grinding of the graphite and gangue.

5. Can be fed direct from jaw-crusher.

It must be stressed that the material submitted is highly weathered. The unweathered portion of the deposit may present quite a different problem.

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Flotation Data

Feed Origin	Rolls, Air Table -10+14 Mesh	Ball Mill 20 min. Grind -14+200 Mesh	Ball Mill 20 min. Grind -200 Mesh	Ball Mill 10 min. Grind -200 Mesh	Ball Mill 20 min. Grind +200 Mesh	Ball Mill 20 min. Grind +200 Mesh	Float #6 Conc. 10 min. Grind Ball Mill 40% Solids	Float #7 Conc. 10 min. Grind Ball Mill 40% Solids	Rolls Product -14 Mesh Large Cell Conc.	Float #9 Conc. 20 min. Grind Ball Mill 70% Solids	Float #9 Conc. 20 min. Grind Ball Mill 40% Solids	Micro-pulverizer Product
Flotation #	1	2	3	4	5	6	7	8	9	10	11	12
Pine Oil lbs/ton	0.37	0.37	0.37	0.37	0.74	0.56	0.22	0.70	-	0.25	0.29	0.74
Kerosene lbs/ton	0.37	0.37	0.37	0.37	0.37	0.27	0.31	0.59	-	0.25	0.59	0.37
NaOH lbs/ton	2.7	2.7	2.7	2.7	0.36	0.31	2.5	2.0	-	1.8	2.1	
Wt. of Sample Gms	149	150	150	150	600	750	174	79.9	704.6	219.4	191.4	600
Conditioning mins.	4	4	4	4	5	5	5	5	5	5	5	5
Tailings Wt. gms	46.7	99.1	118.0	120.6	509.2	500.6	2.3	8.6	242.8	66.7	71.6	250.8
% L.O.I.	Negligible	0.78	1.58	1.45				68.30	17.67	52.48	56.04	
Recovery	-	1.9	9.5	6.9								
Conc. Wt. gms	34.6	42.6	15.2	20.7	129.5	174	79.9	71.5	410.8	152.7	85.5	111.0
% L.O.I.	88.35	91.81	90.09	88.26	93.5			97.1	75.3	83.40	90.80	
Recovery	35.9	93.8	85.5	87.9	73.5			64.4 overall				
Cleaner 1. Tails, Wt. gms	64.8	5.9	9.2	8.2	20.6	46.6	4.2		111.0		34.5	25.7
% L.O.I.	81.67	30.46	10.35	7.24			89.6		37.58		86.70	
Recovery	64.1	4.5	5.2	5.2								
Cleaner 2. Tails, Wt. gms					7.7	60.8						10.5
% L.O.I.												
Recovery												
Cleaner 3. Tails, Wt. gms					6.4							7.5
Recovery												
% L.O.I.												
Cleaner 4. Tails, Wt. gms					4.2							
% L.O.I.												
Recovery												
Cleaner 5. Tails, Wt. gms					3.8							
% L.O.I.												
Recovery												
Water	Distilled	Distilled	Distilled	Distilled	Tap	Tap			Tap	Tap	Tap	Tap
Heads % L.O.I.	56.4	27.6	12.5	15.1	27.6	27.6			51.6	75.5	75.5	22.6