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MINES BRANCH INVESTIGATION REPORT IR 66-92

CALCITE FLOTATION OF PYROCHLORE ORE FROM ST. LAWRENCE COLUMBIUM AND METALS CORPORATION, OKA, QUEBEC

by

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MINERAL PROCESSING DIVISION

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SUMMARY OF RESULTS

Locked-cycle tests were performed on two fine fractions of pyrochlore ore containing large amounts of calcite and assaying 0.43% and 0.35% Nb_2O_3. The rejection of the calcite by flotation from these fine fractions of the pyrochlore ore presented no difficulties. The calcite concentrates, assaying less than 0.1% Nb_2O_5, represent 33% to 52% of the ore by weight and contain between 6% and 12% of the Nb_2O_5 in the fine fraction of the ore. No buildup of deleterious ions was observed and size analyses of the calcite showed that the fine particles and slimes reported in the recleaner concentrates.

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INTRODUCTION

Purpose of Investigation

A revised flowsheet to increase recoveries of niobium at the Oka plant of St. Lawrence Columbium and Metals Corporation would require the flotation of a fine and a coarse fraction of the ore. The flotation of the coarse fraction offers no difficulty. In flotation of the fine fraction, which is all minus 150 mesh, the calcite gangue would be removed by flotation and the calcite float product would then be refloated twice to produce a re-cleaned calcite concentrate with the tailings being returned to the rougher flotation. During pilot plant testing at the Mines Branch, it was discovered that a portion of the finely ground calcite did not float during the refloat step and was returned to the rougher circuit with deleterious effects on the niobium float.

The purpose of this investigation was to conduct locked-cycle flotation tests to determine why the finely ground calcite did not float during the cleaning stages.

Ore Shipment

Mr. J.C. Caron, Vice President of the St. Lawrence Columbium and Metals Corporation, Oka, Que. and Mr. C. Bedard, metallurgist, submitted two ground pyrochlore ore samples on June 8th and 20th, 1966. The ore was ground to minus 150 mesh and was about 70% minus 200 mesh. These samples, designated as "fine fraction of the pyrochlore ore No. 1 and 2", contained 0.43% and 0.27% Nb_2O_5 respectively.

Sample Analysis

All Nb_2O_5 analyses in this investigation were done by the St. Lawrence Columbium and Metals Corporation, Oka, Que.

PROCEDURE AND RESULTS

Three locked-cycle flotation tests, consisting of 5, 10, and 5 cycles were made. All tests were done in a D.R. Denver flotation machine at an impeller speed of 2300 rpm.

The addition of fresh water was kept to a minimum by using the filtrate from the preceding flotation steps. In Cycle Tests 1 and 2, a pilot flotation was done to obtain the required filtrates; in Cycle Test 3, filtrates were kept from Cycle Test 2 and used to start the locked-cycle tests.

The calcite rougher, cleaner, and recleaner flotation stages were done at approximately 30, 15, and 8% solids respectively. The feed for the calcite rougher flotation was composed of the fresh ore, the preceding scavenger float and cleaner tailing, while the calcite cleaner feed consisted of the calcite rougher float and the preceding recleaner tailing.

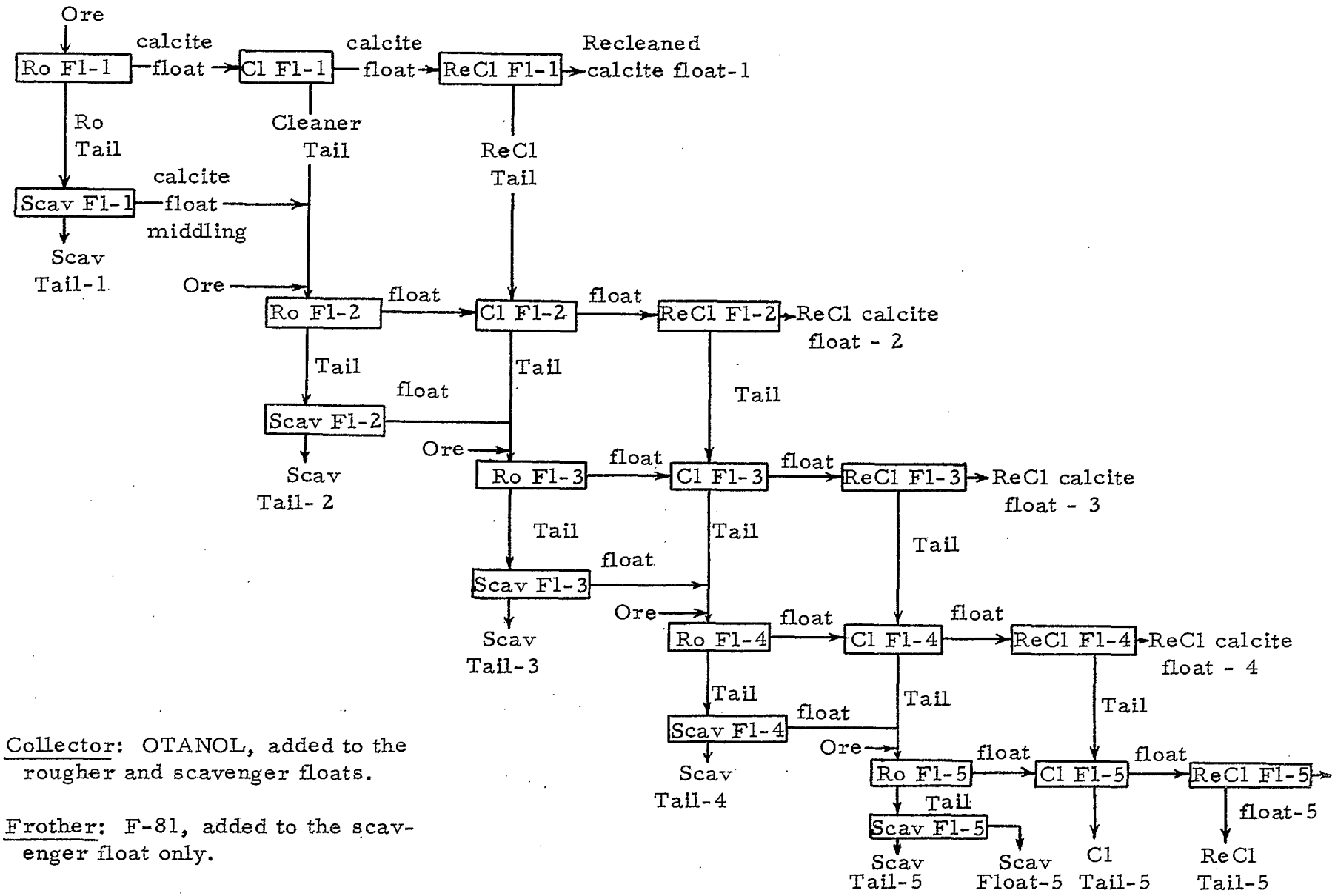
All tests were done at the natural pH of the ore, 8.8 - 9.0. A fatty acid product called Otanol (produced by Tevasaari Pulp and Paper Mill, Finland and distributed by Kingsley and Keith, Canada, Ltd.) was used as the collector for calcite. The Otanol was fed as a 1% emulsion, at a rate of 0.5 to 0.6 lb/ton, to the calcite rougher feed and 0.1 lb/ton to the scavenger feed. About 0.01 lb of frother F-81 per ton was added to the scavenger float only. The return water carried enough frother from the pilot test for the rougher float in the first cycle. Both reagents were conditioned in the flotation cell for 5 minutes before air introduction. No depressant for the pyrochlore was used in these tests.

Cycle Test 1

Cycle Test 1, consisting of five cycles was carried out as described in the general procedure and Figure 1. The filtrates from previous flotation tests were used for the repulping of all succeeding calcite floats.

Prior to Cycle Test 1, a pilot test using fresh water for repulping the calcite floats was made to obtain filtrates to start the Cycle Test and keep conditions standard.

Filtrates were used instead of fresh water to repulp the floats to determine if any buildup of slimes or deleterious ions occurred to create problems in the succeeding cycles. The ore used in this Cycle Test consisted of ore No. 1 with a head assay of 0.43% Nb_2O_5.



Collector: OTANOL, added to the rougher and scavenger floats.

Frother: F-81, added to the scavenger float only.

Figure 1. Five cycle flowsheet used in locked-cycle flotation tests.

The results of this Cycle Test are recorded in Table 1, which also includes the pilot test designated as zero (0).

TABLE 1

Results of Cycle Test 1, Including Pilot Test, On Ore No. 1 With Head Analysis of 0.43% Nb_2O_5

	Products	% Weight	% Nb_2O_5 Assays	% Nb_2O_5 Distribution
Pilot Test	Scav Tail - 0	62.6	0.605	87.1
	Scav Float - 0	7.3	0.155	2.6
	Cl Tail - 0	9.5	0.260	5.7
	ReCl Tail - 0	3.1	0.170	1.2
	ReCl Float - 0	17.5	0.085	3.4
	Flotation Feed	100.0	0.435	100.0
Cycle Test 1	Scav Tail - 1	10.7	0.745	18.2
	Scav Tail - 2	9.1	0.805	16.6
	Scav Tail - 3	9.7	0.850	18.9
	Scav Tail - 4	8.8	0.775	15.5
	Scav Tail - 5	9.3	0.830	17.5
	Scav Tail 1 - 5	47.6	0.799	86.7
	Scav Float - 5	1.3	0.240	0.7
	Cl Tail - 5	3.8	0.250	2.0
	ReCl Tail - 5	1.8	0.215	0.9
	Middlings	6.9	0.230	3.6
	ReCl Float - 1	7.5	0.080	1.4
	ReCl Float - 2	9.1	0.095	2.0
	ReCl Float - 3	9.1	0.090	1.8
	ReCl Float - 4	10.2	0.095	2.3
	ReCl Float - 5	9.6	0.095	2.1
	ReCl Float 1 - 5	45.5	0.093	9.6
	Flotation Feed	100.0	0.439	100.0

Otanol addition: 0.50 lb/ton to the rougher float and 0.10 to the scav.float
F-81 addition : 0.01 lb/ton to the scavenger float only.

Effect of Ca, Mg ions and $CaCO_3$ as Total Hardness, on Flotability of Fine Calcite

To determine if there was a buildup of deleterious ions which could prevent the fine calcite from floating in the subsequent floats of the cycle test, filtrate samples of the recleaner floats were analyzed for Ca, Mg ions and $CaCO_3$ as total hardness. As shown in Table 2, there was no buildup of these ions and the good results of these flotation tests as shown in Table 1, indicated that the fine calcite refloated properly and reported in the calcite recleaner floats. Analyses of the filtrates of the scavenger tails from cycles 0 and 5 showed a slight buildup of ions but again the flotation results indicate that this slight increase caused no problem.

TABLE 2

Results of Analysis of Filtrates to Determine Concentration of Ca, Mg ions and $CaCO_3$ as Total Hardness in PPM

Products	PPM in Filtrates		
	Ca	Mg	$CaCO_3$
Scav Tail - 0	18.9	3.32	60.9
Scav Tail - 5	23.7	4.96	79.6
ReCl Float - 1	23.0	4.32	75.2
ReCl Float - 2	22.4	4.74	75.5
ReCl Float - 3	20.8	4.74	71.5
ReCl Float - 4	24.3	4.88	80.7
ReCl Float - 5	22.4	4.76	75.5
Composite	22.2	4.78	75.1

Size Analysis of Flotation Products

Particle size analyses were made on 3 calcite recleaner floats and 3 scavenger tailings of cycles 0 (pilot test), 1 and 5 and on the feed (ore No. 1) to Cycle Test 1, to determine if the amounts of fine particles and slimes varied in the different products throughout the Cycle Test. A sedimentation process using a hydrometer according to ASTM method

D 422-54T was chosen to obtain the necessary data. In choosing this method, the assumption was made that the ore was homogeneous, a reasonable assumption since the ore contains only 0.43% Nb_2O_5; the specific gravity of pure calcite is 2.72 compared to 2.83 for the ore. Also, this was the only method available at the time to determine sizes below 38 microns.

The curves in Figure 2 indicate that there is a slight decrease in the amount of fines in the scavenger tailings from cycles 0, 1 and 5 and also a decrease in the amount of minus 10 micron particles in the flotation products from the feed. Figure 3 indicates that the finer particles are concentrated in the calcite recleaner products, although there is an inversion in the curves of cycles 1 and 5. These curves would then seem to indicate that the fine particles float in the recleaner stages and do not accumulate in the scavenger tails to affect the flotation of pyrochlore, later.

Cycle Test 2

The flotation feed for this Cycle Test was the same ore, No. 1, as used in Cycle Test 1. Cycle Test 2 consisted of a pilot test and ten cycles to confirm data obtained in Cycle Test 1 and to observe if any changes would occur by including an extra five cycles. The results as recorded in Table 3 incorporate the pilot test as cycle 0. The same procedures were used as described for Cycle Test 1 with the exception that five extra cycles were added.

Cycle Test 3

The flotation feed for this Cycle Test 3 was composed of equal amounts by weight of ores No. 1 and 2 with a head assay of 0.35% Nb_2O_5. This was felt to be a more representative grade of the ore presently used at Oka. All tests were done as described previously for Cycle Tests 1 and 2, but without a pilot test. Filtrates from Cycle Test 2 were used to pulp the ore in the first cycle. The results are presented in Table 4.

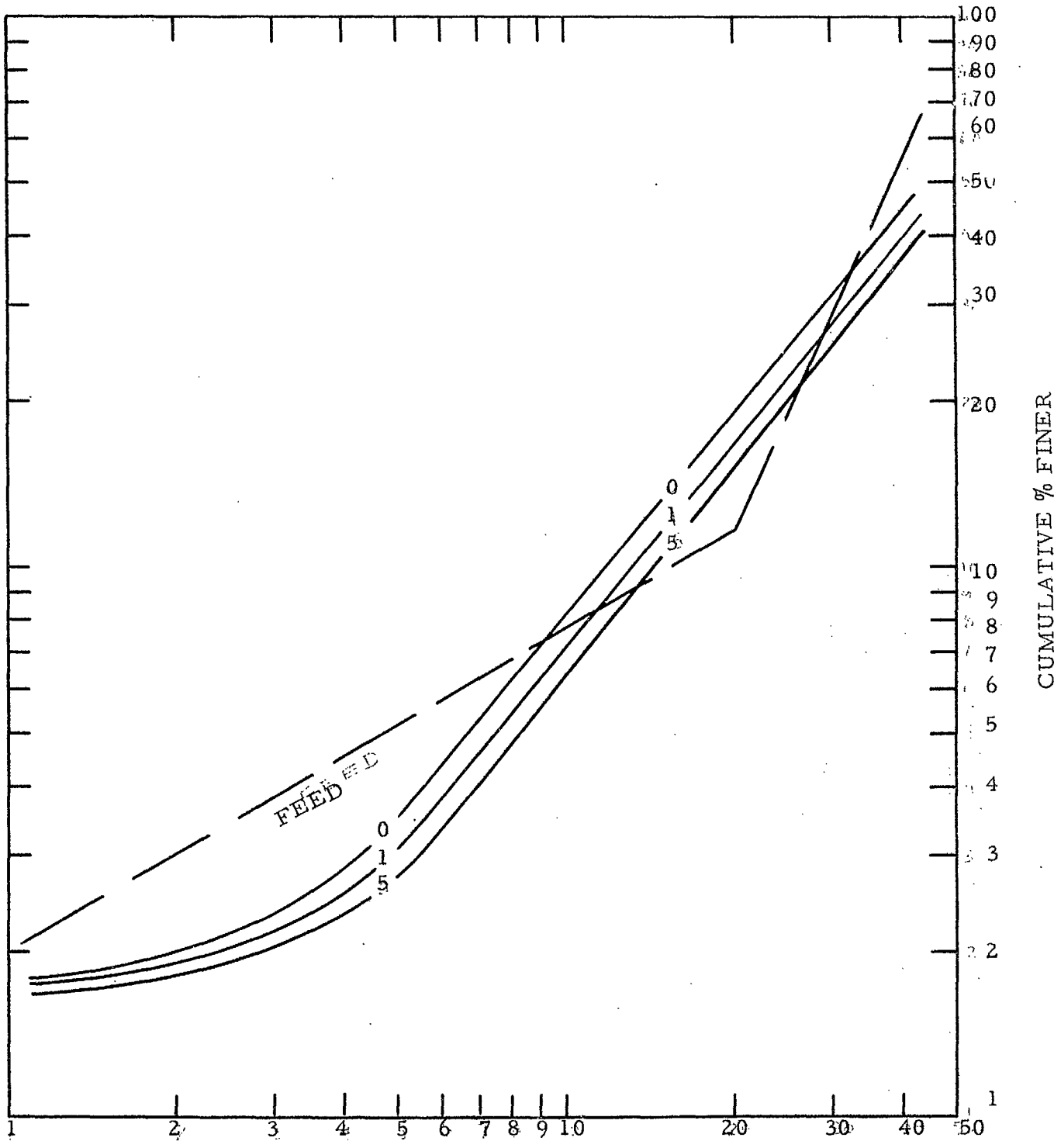


Figure 2. Size analysis of feed and scavenger tails of cycles 0 (pilot test), 1, and 5 of Cycle Test 1, by sedimentation.

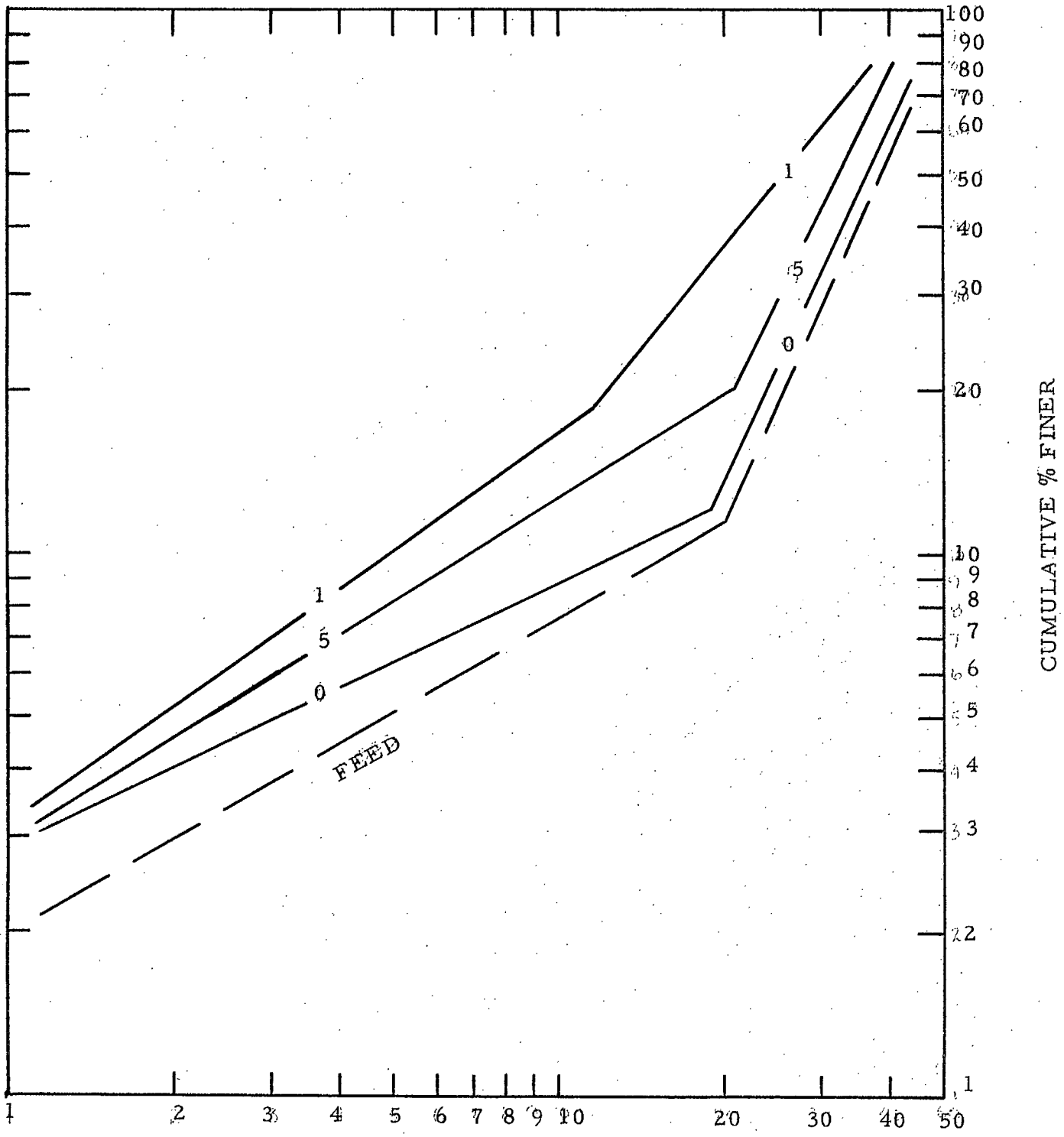


Figure 3. Size analysis of feed and recleaner floats of cycles 0 (pilot test), 1, and 5 of Cycle Test 1, by sedimentation.

TABLE 3

Results of Cycle Test 2, Incorporating Pilot Test, on Ore No. 1 With Head Analysis of 0.43% Nb_2O_5

Products	% Weight	% Nb_2O_5	
		Assays	Distribution
Scav Tail - 0	6.0	0.600	8.3
Scav Tail - 1	6.5	0.570	8.7
Scav Tail - 2	6.2	0.570	8.2
Scav Tail - 3	6.0	0.620	8.6
Scav Tail - 4	5.7	0.620	8.2
Scav Tail - 5	5.7	0.610	8.1
Scav Tail - 6	5.6	0.630	8.2
Scav Tail - 7	5.8	0.680	9.1
Scav Tail - 8	5.5	0.680	8.7
Scav Tail - 9	5.3	0.680	8.2
Scav Tail -10	5.1	0.690	8.2
Scav Tail 0-10	63.4	0.630	92.5
Scav Float -10	0.8	0.170	0.3
Cl Tail -10	1.6	0.280	1.0
ReCl Tail -10	0.7	0.210	0.4
Middlings	3.1	0.240	1.7
ReCl Float - 0	0.9	0.055	0.1
ReCl Float - 1	0.8	0.080	0.1
ReCl Float - 2	3.9	0.095	0.8
ReCl Float - 3	3.5	0.070	0.6
ReCl Float - 4	3.3	0.065	0.5
ReCl Float - 5	3.4	0.075	0.7
ReCl Float - 6	3.1	0.060	0.4
ReCl Float - 7	3.3	0.075	0.6
ReCl Float - 8	3.9	0.070	0.6
ReCl Float - 9	3.9	0.080	0.7
ReCl Float -10	3.5	0.075	0.7
ReCl Float 1-10	33.5	0.074	5.8
Flotation Feed	100.0	0.431	100.0

Otanol addition: 0.48 lb/ton to the rougher float and 0.08 to the scav. float.

F-81 Addition : 0.01 lb/ton to the scavenger float.

TABLE 4

Results of Cycle Test 3 on Equal Amounts of Ore No. 1 and 2 With Head Assay of 0.35% Nb_2O_5

Flotation Products	% Weight	% Nb_2O_5 Assays	% Nb_2O_5 Distribution
Scav Tail -1	9.8	0.675	18.8
Scav Tail -2	8.2	0.710	16.6
Scav Tail -3	7.2	0.800	16.6
Scav Tail -4	7.4	0.770	16.3
Scav Tail -5	7.4	0.880	18.6
Scav Tail 1-5	40.0	0.760	86.9
Scav Float -5	1.8	0.155	0.8
Cl Tail -5	3.7	0.285	3.0
ReCl Tail -5	1.9	0.201	1.1
Middlings	7.4	0.232	4.9
ReCl Float -1	7.7	0.045	1.0
ReCl Float -2	11.2	0.050	1.6
ReCl Float -3	11.0	0.055	1.8
ReCl Float -4	11.3	0.060	1.9
ReCl Float -5	11.4	0.060	1.9
ReCl Float 1-5	52.6	0.054	8.2
Flotation Feed	100.0	0.350	100.0

Otanol addition: 0.52 lb/ton to the rougher float and 0.10 lb/ton to the scavenger float.
F-81 addition : 0.01 lb/ton to the scavenger float only.

DISCUSSION OF RESULTS

The results from the three laboratory Cycle Tests (Tables 1, 3, 4) show that the rejection of the calcite from the fine fraction of the pyrochlore ore offers no difficulty. The amount of calcite concentrate and the Nb_2O_5 grade which is rejected seems to depend only on the consumption of collector for the calcite. In Cycle Test 1, for an Otanol consumption of 0.60 lb/ton, 0.09% Nb_2O_5 recleaner calcite concentrate was floated representing 46% of the weight compared to a 0.07% Nb_2O_5 recleaner calcite concentrate representing 34% of the weight for an Otanol consumption of 0.56 lb/ton for Cycle Test 2. Using a different ore with a lower head assay in Cycle Test 3, a 0.05% Nb_2O_5 recleaner calcite concentrate was obtained, representing 53% of the weight for an Otanol consumption of 0.62 lb/ton. The niobium losses to this calcite concentrated were 10%, 6% and 8% respectively. The individual assays and weight rejections for each cycles in the three Cycle Tests were relatively constant except for the pilot tests.

The grade of the middling products is surprisingly constant at 0.23% to 0.24% Nb_2O_5 although its weight distribution varies.

The grades and recoveries of Nb_2O_5 in the scavenger tailings also are proportional to the amount of collector used to float the calcite and represent good results: 0.8% Nb_2O_5 with 87% recovery in Cycle Test 1, 0.7% Nb_2O_5 with 93% recovery in Cycle Test 2 and 0.8% Nb_2O_5 with 87% recovery in Cycle Test 3.

From these results only, it would then seem that the fine calcite is floating well and responding to collector consumption, and its floatability is not affected by deleterious ions and slimes.

From Table 2, the concentration of Ca and Mg ions and $CaCO_3$ stays relatively constant and therefore a buildup of these noxious ions does not seem to be the cause of the difficulties encountered in the pilot-plant run.

It was observed that the calcite concentrates had a much lower filtration rate than the scavenger tailings. In addition, the filtration rate of the calcite concentrate seemed to decrease as the number of tests in a Cycle Test increased. This phenomenon thus indicated that fine calcite was accumulating in the calcite concentrate where it belonged and not in the scavenger tailings. These observations are born out by the size analysis curves presented in Figures 2 and 3.

CONCLUSIONS

The difficulties encountered in the pilot plant run could not be explained by the results of this investigation. There do not appear to be any physical or chemical characteristics of the ore causing the lack of flotation of slimes or fine particles in the cleaning stages. Other possible causes of this problem can be investigated only in a pilot plant run. It might therefore be advisable to make another pilot plant run to investigate these causes before any additional research is instituted.

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