RECOVERY OF FELDSPAR AND QUARTZ FROM SPRUCE PINE FILTER CAKE WASTE

by
John G. Groppo, Jr.
Mineral Engineer

ABSTRACT

Extremely fine-ground ceramic-grade feldspar can be recovered from IMC filter cake waste in batch flotation tests. A small laboratory hydrocyclone was used to deslime the feed so that material slightly finer than 12 microns could be recovered. Recovery and selectivity were improved by reducing both turbulence in the cell and the shear forces acting on the particles during flotation.

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INTRODUCTION

A composite sample of filter cake waste produced by IMC in Spruce Pine, North Carolina, was collected over a one-week period. Flotation tests were conducted to recover any feldspar and quartz contained in the waste. The extremely fine size distribution of this material necessitated the development of several specialized techniques particularly in the desliming phase. Flotation tests were conducted under a variety of operating conditions to determine the parameters necessary to obtain optimum results.

The feldspar contained in this type of waste material was investigated by the Minerals Research Laboratory in Asheville, NC, in a report published in September 1969. This report suggested that glass could be produced from the filter cake tailings as well as sand-lime bricks and possibly ceramic whiteware.

SAMPLE PREPARATION

A composite sample was collected from the tailings disc filter during the week of June 23, 1980. Approximately ten pounds of filter cake material was collected every day while the plant was operating to formulate a representative sample of the filter cake tailings. This composite sample was then turned over to the Minerals Research Laboratory for testing.

The composite sample had a moisture content of 21.6% upon receipt, and the material was made up almost entirely of fine particles packed together into lumps. In order to thoroughly mix this composite sample, it was necessary to break up all of these lumps. To accomplish this, the entire sample was dried for 24 hours at 200° F and allowed to cool. Once cooled, the entire sample was spread onto a large sheet of plastic so that the lumps could be broken by rolling a large piece of plastic pipe over the sample. The material was screened on a US 20 screen, and the oversize was crushed again until it passed through the US 20 screen.

Once the sample was of a fairly uniform size, it was possible to mix it thoroughly and obtain 500-gram samples for testing. Size distribution (Table 1) and mineral content (Table 2) were then determined.

TABLE 1

SIZE DISTRIBUTION AND ANALYSES OF FILTER CAKE MATERIAL

			Chemical Assays			
US Screen	Wt %	Cum. Wt % Pass.	K_2O	Na_2O	CaO	Fe_2O_3
+60	1.7	98.3	6.82	2.52	0.82	3.60
-60+100	4.1	94.2	5.28	3.52	0.82	1.92
-100+140	6.1	88.1	4.52	4.02	0.98	1.23
-140+200	12.8	75.3	4.08	4.28	1.11	0.81
-200+325	39.8	35.5	3.70	4.30	1.96	0.81
-325+400	16.3	19.2	3.54	4.08	4.14	1.10
-400	19.2	0	3.54	4.02	4.14	1.06
Total	100.0	-	3.86	4.13	2.50	1.02
Head Sample	-	-	3.84	4.08	2.51	1.86

TABLE 2

MINERAL CONTENT OF SAMPLE 4731-B

Wt %	Mineral
32.7	Soda Feldspar
19.0	Potash Feldspar
9.2	Lime Feldspar
60.9	Total Feldspar
4.0	Mica
4.7	Kaolin
30.4	Quartz and Iron Minerals

TESTING PROCEDURE

Because this material was quite fine (88% minus 100 mesh), there was no need for additional comminution. The sample did contain some clay material which would have to be removed in order to employ flotation techniques for mineral separation.

Each sample was scrubbed for 5 minutes at 70% solids with 1.0 lb/ton NaOH. The scrubbed sample was then deslimed to remove the clay minerals. Desliming techniques will be discussed later in this report. Adequate desliming is essential for flotation of this type of ore and was the subject of an involved study aimed at improvement of desliming techniques.

To reduce the iron content of the ore, high-intensity wet magnetic separation (HIW) was used. Several tests using sulfonate floats were attempted, but these did not reduce the iron content to the desired levels.

Conditioning was accomplished in a 1000-ml beaker using a stainless steel, 4-bladed impeller which rotates at 700 rpm. Denver flotation machines were used for the flotation separations. Due to the extremely fine nature of the material being floated, several changes were made in the operation of the flotation machines to achieve optimum results. These changes will also be discussed later in this report. A summary of several of these flotation tests is presented in Table 3.

RESULTS

Comparison of Tests 10 and 13 presented in Table 3 indicates that both settling and hydrocycloning are effective techniques for desliming. The results obtained by these two tests were nearly identical.

Sulfonate floats for iron removal were not as effective as high-intensity wet magnetic separation (HIW), although the sulfonate float did yield a feldspar product with a much lower free-quartz content.

Feldspar recovery of as high as 98.8% was obtained by increasing the amine consumption to 1.5 lb/ton in Test 19, while a recovery of nearly 98% was obtained in other tests using only 0.8 lb/ton of amine. The size distribution of the feldspar flotation product is presented in Table 4, which indicates that approximately 65% of the product is finer than 37 microns (-400 mesh). A cleaner float was performed on each feldspar float product, with the small quantity of middling being combined with the quartz tailings for assay.

Mica floats were also cleaned once. Optimum amine consumption was 0.15 lb/ton, yielding a product that was quite high in grit content. Most of the biotite was removed by HIW.

Several non-HF feldspar flotation tests were also completed; the results being presented in Table 5. High recovery and fair selectivity appear to be characteristic of this float, but additional study in this area is necessary.

SPECIFIC PROBLEMS

Desliming is a critical stage in any amine flotation process. The most frequently employed desliming technique for batch flotation tests is settling. The sample is generally slurried in a full bucket of water, allowed to settle for a specified length of time and decanted through a screen to check on the efficiency of the procedure. This method is adequate for most materials that are studied, but the extremely fine

TABLE 3

FLOTATION TESTS WITH LAB 4731-B

Test No.	Desliming Method	Iron-Mineral Removal	Mica Flotation: Armac-T[1] (lb/T)	Mica Flotation: Flot.Speed (rpm)	Spar Flotation: Armac-T (lb/T)	Spar Flotation: HF[2] (lb/T)	Spar Flotation: Flot.Speed (rpm)	Feld.Recovery (%)	Free Qtz. in Spar	Fe_2O_3 Analyses: Spar	Fe_2O_3 Analyses: Quartz	Additional Air
3	Settling	HIW & Flot.[4]	0.15	1200	0.7	1.5	1200	88.4	8.7	0.20	0.005	No
7	Settling	HIW	0.20	1200	0.7	1.5	1200	78.9	2.6	0.26	0.110	No
8	Settling	HIW	0.20	900	0.7	1.5	900	98.0	11.0	0.036	0.011	No
9	Settling	HIW	0.10	900	0.7	1.5	900	97.8	5.7	0.090	0.022	No
10	Settling	HIW	0.15	900	0.7	1.5	900	97.6	8.7	0.088	0.014	Yes
13	Cyclone	HIW	0.15	900	0.7	1.5	900	97.8	10.3	0.067	0.010	Yes
14	Cyclone	HIW	0.15	900	0.8	1.5	900	97.8	13.3	0.071	0.010	Yes
15	Cyclone	HIW	0.15	900	0.8	1.5	900	98.1	14.4	0.063	0.010	Yes
16	Cyclone	HIW	0.10	900	0.8	1.5[3]	900	94.2	11.4	0.110	0.140	Yes
17	Cyclone	HIW	0.125	900	0.8	1.5	900	98.5	10.4	0.059	0.140	Yes
18	Cyclone	Flotation[4]	-	-	0.8	1.5	900	98.7	1.5	0.091	0.018	Yes
19	Cyclone	HIW	0.125	900	1.5	1.5	900	98.8	8.9	0.059	0.010	Yes
20	Cyclone	Flotation[4]	-	-	0.8	1.5	900	98.0	2.2	0.121	0.027	Yes

(1) Armac-T = amine acetate, Armak Company, 300 South Wacker Drive, Chicago, Illinois 60606

(2) HF = hydrofluoric acid, Fisher Scientific Company, 711 Forbes Ave., Pittsburgh, PA 15219

(3) H_2SO_4 instead of HF

(4) HM-70, used in iron float = Morco HM-70, Marathon Morco Company, Dickinson, Texas 77539

TABLE 4

SIZE DISTRIBUTION OF FELDSPAR FROTH PRODUCT

TEST 19

Size (microns)	% Weight	Cum. Weight % Pass.
+297	1.0	99.0
-297+149	2.5	96.5
-149+74	14.4	82.1
-74+42.5	15.2	66.9
-42.5+30.7	9.8	57.1
-30.7+22.7	21.3	35.8
-22.7+15.4	16.2	19.6
-15.4+11.9	5.6	14.0
-11.9	14.0	0

TABLE 5

NON-HF FLOTATION RESULTS

Test No.	TDO* (lb/ton)	Spar Recovery %	Free Quartz %
43	0.90	97.2	15.2
41	1.00	93.4	10.5
42	1.25	96.8	19.7
40	1.50	98.1	21.6

*TDO = Duomeen TDO, Armak Company, 300 South Wacker Drive, Chicago, Illinois 60606 nature of Lab 4731-B presented several problems. It was desired to recover as much as possible of the feldspar contained in the waste. This meant that settling time would be in excess of 25 minutes before decanting, and it would be necessary to do this twice. To remedy this problem, a different desliming technique was developed. This technique employed a small hydrocyclone which was operated so as to deslime the feed at 12 microns. This desliming procedure simulates operating conditions which would be encountered in production. Details of this desliming technique are available in MRL Report 80-15-P.

Another specific problem was encountered in the flotation phase. Standard laboratory equipment is not designed to effectively handle material that is in the sub-sieve size range. Normally, flotation experiments are carried out at 1200 rpm, and high selectivity can be obtained by using proper levels of reagents. By experimentation, it was found that higher selectivity can be obtained with sub-sieve size particles when the flotation machine is operated at 900 rpm. This slower speed significantly reduces the shear forces acting on the particles and drastically reduces turbulence in the cell. It is thought that the greater shear forces and turbulence caused by operation at 1200 rpm cause extremely fine particles to be carried to the cell surface by action other than attachment to bubbles. This results in poor selectivity even when reagent levels are in proper proportion. The slower machine speed reduces turbulence in the cell so that particles may be brought to the cell surface by attachment to air bubbles, not by mechanical agitation.

When operated at 1200 rpm or above, the Denver batch flotation machines generate sufficient air bubbles to float the desired particles. They do not generate enough air bubbles at slower speeds, where more air is needed. Equal weights of fine material and coarse material do not have the same surface area, the total surface area of the fine material is much greater. Realizing this, it stands to reason that more bubble surface would be required to float the material containing more surface area. That is why it became necessary to add air to the flotation machines while they operated at a slower speed.

A compressor was used to force small quantities of air into the air valve on the standpipe of the flotation machine. This generated adequate air to float all of the desired particles. Without this additional air, the floated particles formed a tenacious froth in the center of the cell that required vigorous paddling to be skimmed off. This vigorous paddling decreased the selectivity of the float by inadvertently skimming off particles that were not actually floated. Due to the close tolerance between the flotation machine standpipe and impeller shaft, forced air was not dispersed throughout the entire cell. The compressed air entered the air valve and was dispersed at the base of the impeller directly below the valve (Figure 1), giving aeration that only affected half of the cell. There was no aeration in the other half of the cell. Inserting an additional air valve into the standpipe provided an increase in the aeration throughout the cell, although it was still necessary to use compressed air. The net effect was a more dispersed aeration. The ideas presented in Figures 1 and 2 are idealized explanations of the air flow restrictions of the Denver batch flotation machines. These figures represent only a partial explanation of the air flow patterns as they were observed during flotation tests.

DISCUSSION

The net result of reducing impeller speed and increasing air flow is a better-controlled float where the froth product can be skimmed off without inadvertently pulling undesired material. This provides a much more reproducible float with higher recovery. Referring to Table 6, it is evident that recovery is increased from 78.9% to 98.0% simply by reducing the impeller speed. The free-quartz content of the feldspar product in Test 8 is significantly higher, but this is probably due to vigorous paddling that was required since neither of these tests utilized additional air. There is no explanation for the much lower Fe_2O_3 content of the feldspar product obtained at the slower speed.

The free-quartz content of the feldspar product is significantly lower when sulfonate flotation rather than HIW is employed for iron removal. This is due to the excessive amount of fine material other

CROSS SECTION OF DENVER BATCH FLOAT CELL

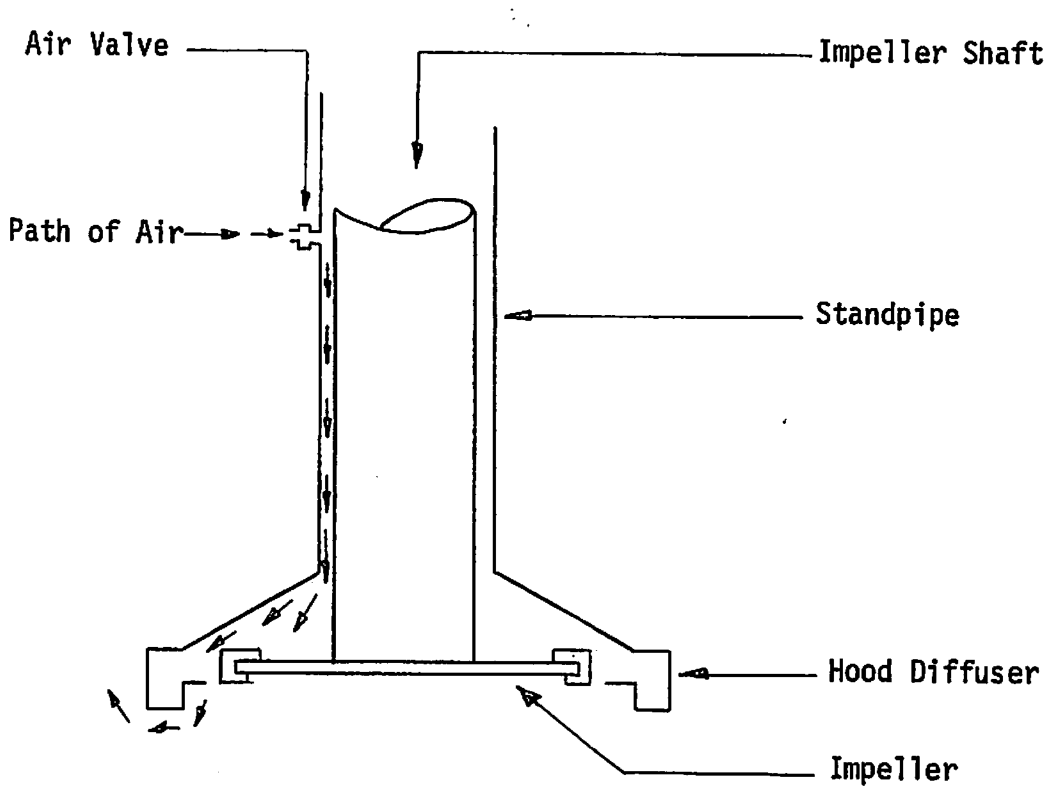

Figure 1

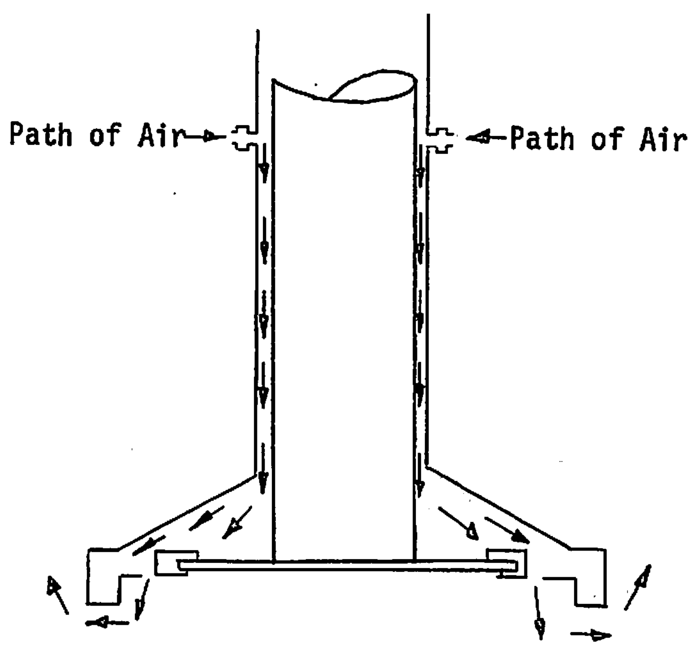

Figure 2

than iron-bearing minerals that is floated in the sulfonate float. Most of this excess material is quartz, which explains why there is much less quartz in the feldspar product.

The extremely fine size of this waste material should make it particularly suitable for non-HF flotation,which is known to give good results with minus 35 mesh material. However, from the data presented in Table 5, the selectivity of the non-HF float is much poorer than that of the standard HF float , while the recoveries of both tests are similar. Since there has been limited research on the application of the non-HF float to extremely fine material, an additional study should be considered.

TABLE 6

COMPARISON OF CHANGING IMPELLER SPEED

	Test 7	Test 8
High Intensity Wet Magnetic Separation	4 passes	4 passes
Mica Float - Amine Quantity (lb/ton)	0.2	0.2
Spar Float - Amine Quantity (lb/ton)	0.7	0.7
Weight % Spar F.P.	58.5	63.5
rpm of Flotation Impeller	1200	900
% Recovery of Feldspar	78.9	98.0
% Free Quartz in Feldspar	2.6	11.0
Fe_2O_3 Content of Feldspar	0.26	0.036

CONCLUSIONS

Batch testing indicates that IMC filter cake waste material contains a large quantity of fine feldspar and quartz that can be recovered. High-intensity attrition scrubbing, adequate desliming, and proper float cell design are critical parameters to which attention must be directed. Recovery of waste material is of valid environmental and economic concern; conversion of waste into salable products would reduce waste disposal costs while generating additional revenue. Mining and comminution costs are eliminated because the material is a plant by-product that has already been completely liberated.

Coarse feldspar is difficult to float in the non-HF system; since there is no coarse feldspar in the waste material, the non-HF procedure should be particularly suitable. Iron removal by flotation seems doubtful if sufficient grade and optimum recovery are to be achieved. High intensity wet magnetic separation is recommended for iron removal.

SUGGESTIONS

Based upon the data presented in this report, the following areas of future research can be suggested:

1) Plant sampling to determine the cause of such high feldspar content of the present waste material.

2) Study of application of non-HF flotation of feldspar to achieve results that are more comparable with HF flotation.

3) Desliming at sizes finer than 12 microns when adequate sizing equipment is available.

4) Further investigation of mica flotation parameters to obtain an acceptable mica product.

5) Testing of the feldspar and quartz products to determine their possible future markets.

6) Eventual pilot plant study with filter cake material as feed.

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