BENEFICIATION OF FELDSPAR FILTER CAKE TAILINGS

Feldspar Tailings Report No. 4, April 1969 Progress Report
Lab. Notebook No. 236 - Lab. Sample No. 3300
by

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Background of Test Work

During the summer of 1968 the Minerals Research Laboratory was in the process of ascertaining the type, composition, and quantity of the tailings coming from the plants of the three feldspar-producing companies in the Spruce Pine area. This project was being undertaken under contract with the U. S. Bureau of Mines and the three companies.

One of these tailings is filter cake product which is substantially minus 200 mesh. It was found desirable to determine the amount of recoverable feldspar and quartz (glass or ceramic grade) in this fine material. Accordingly, a research program was set up with the hope of determining how much of each was actually available and employing some means of separation which appeared economically practicable.

Sample No. 3300 - Collection, Preparation, and Screen Analysis

The filter cake tailings product of International Minerals and Chemical Corporation was selected as representative of potential problems and potential values involved. The sample was collected during the first week of July 1968 from the foot of the discharge belt carrying hydroclassified filter cake fines to the yard outside the IMCC tailings plant. The total sample collected weighed about 600 pounds after drying. Moisture content on receipt was 23 percent.

It was assumed that most effective beneficiation could be obtained after scrubbing to remove the various reagents present, and to aid in creating fresh particle surfaces. In order to maintain better control of scrub pulp density, a dry feed sample was preferred, and so the entire sample was dried at 250°F.

Screen analysis of Sample No. 3300 as received differed to a slight degree from a composite of this filter cake assembled over a period of months, but was judged similar enough to offer representative problems of beneficiation. Table 1 shows screen analysis of No. 3300 compared with the long-range composite, which latter is also referred to in Table 3 of Feldspar Tailings Report No. 3, April 1969 Progress Report.

Direction of Research

Among the many unknowns regarding beneficiation of this filter cake was the question as to how fine a particle size was still amenable to fairly standard flotation procedures used to concentrate mica, iron minerals, feldspar, and quartz. It was decided to give principal attention to this question. Therefore, conditions of scrubbing, desliming, hydroclassification, and screening - followed by conditioning and flotation - were set up with this in mind.

Scrubbing, Desliming, and Hydroclassification

A few preliminary tests disclosed that a scrub pulp density of 72 percent solids, with 2 pounds per ton of $NaOH$, was close to the maximum thickness permitting good pulp circulation, and these conditions were maintained for all scrub procedures. Scrub tests in the beginning were carried out on 1000-gram batches of Sample No. 3300, but it was considered feasible to switch to a large batch scrubber and process 13 kilograms at a time. Scrub duration was 10 minutes for 1000 grams and 15 minutes for 13 kilograms. In both cases the scrubbed pulp reached a temperature of about 150°F.

Scrub apparatus was as follows: for 1000-gram batches, a Wemco Mineral Master 1000-gram assembly consisting of octagonal pot and 3-tiered impellers rotating at 1350 rpm; and for 13 kilogram batches, a similar machine with an octagonal pot measuring 9 1/2 inches between opposite parallel sides, and a 3-tiered impeller assembly with blades 8 5/8 inches in diameter, turning at 475 rpm.

Desliming and hydroclassification following scrubbing were aimed at accurate and definitive size classification, rather than at demonstrating optimum procedure or equipment in a theoretical flowsheet. Plus 200 mesh ore was eliminated by classifying and screening. The remainder was split into progressively finer fractions by settling for various times and decanting. The response of these fractions to flotation was determined.

Initial classification tests were carried out on 1000-gram portions, and after removal of the plus 200 mesh fraction ("coarse"), further classification was carried out to yield "intermediate," "fine," and "slimes" fractions. Table 2 gives weight percentages and screen analyses of these products. As flotation tests went forward, it became apparent that it was feasible to combine the "intermediate" and "fine" fractions. Thus the number of classified products was reduced to three: "coarse," "fine," and "slimes." Also, to enable the running of more tests, the procedure to scrub and hydroclassify 13 kilos of filter cake at a time was worked out, using larger apparatus. Table 3 gives screen and weight ratio characteristics of the "fine" fraction from Test No. 30 (13 kilo batch) compared to the combination of "fine" plus "intermediate" fractions from a typical 1000-gram batch classification (Test No. 5). Details of scrubbing and hydroclassifying procedure can be found in Laboratory Notebook No. 236.

Conditioning and Flotation Procedure

A USBM-type flotation of the mica present in the "fine" fraction gave quite satisfactory visual results in selectivity and removal. However, it was judged more important to spend the time available on research connected with feldspar recovery. Fewer procedures prior to feldspar concentration would cut down time, and reduce possible variables which might invalidate tests. Therefore, an acid-circuit petroleum sulfonate float was employed as a single scavenger step to remove both mica and iron minerals. Following this was a standard feldspar float employing HF and tallow amine acetate.

Standard procedure in the conditioning and float tests described was as follows: 13 kilograms of Sample No. 3300, dried, was scrubbed and classified as previously described and tests run on 500-gram aliquots of the "fine" fraction. Conditioning at 65 percent solids was performed with a conditioner having a single tier of 45 degree blades, running at 700 rpm. The first conditioner step used the reagents H_2SO_4, petroleum sulfonate (M-70), and pine oil. Flotation for iron minerals was carried out in a 1000 gram Denver D-1 Lab Cell at 1200 rpm. Following flotation, the machine discharge was dewatered; re-diluted in the cell with fresh water; and conditioned at about 40 percent solids with HF, tallow amine acetate, fuel oil, and pine oil. Conditioning without air was for 5 minutes, followed by aeration and flotation - all at 1200 rpm.

Variables and results of the most significant tests are shown in Table 4. Table 5 gives characteristics of the products of one of the tests shown in Table 4 (Test No. 61). In all these tests, H_2SO_4 level varied between 1.6 and 2.5 lbs per ton, calculated against 500 grams of "fine" fraction, but in Table 4 only conditioner pH is cited. Pine oil was kept constant at about 0.1 lbs/T in the first conditioner and about 0.2 lbs/T in the second-calculated in the same way (1 drop and 2 drops, respectively). Fuel oil in the second conditioner - float cycle was constant at about 1.0 lbs/T (10 drops), and HF was constant at 1.5 lbs/T, again based on the "fine" fraction.

In addition to evaluation of the feldspar product of Test No. 61 through screening, chemical analysis, and average particle size determination, a sub-sieve size distribution curve was established and compared with that of a typical fine dry-ground feldspar sold for ceramic and filler use. This curve is shown in Figure 1. The tests were run by hydrometer according to ASTM procedure D-422-61T. Table 6 gives further comparative physical and chemical data of the feldspar product from Test No. 61 and a typical fine-ground ceramic feldspar.

<u>General Information Developed</u>

Surveying some preliminary tests, plus the data of Tables 4, 5, and 6, the following seems apparent:

(1) It is possible to successfully perform conditioning and flotation upon a scrubbed and sized fraction of filter cake as Sample No. 3300, which is essentially all minus 200 mesh, and 75 percent minus 400 mesh, employing techniques and conditions basically the same as with 20 - 200 mesh ore of like composition. Principal variations required are length of conditioning time and collector level (in both floats), all of which seem to need increase by a factor of about 2.

(2) The main flotation product - feldspar - has essentially the screen size characteristics of sanitary ware - or latex filler-grade spar, and also close to the same chemical analysis, except for slightly high Fe_2O_3, regarding which further research may well find an answer. The much coarser average particle size of this spar - compared to the usual dry-ground product - indicates needed investigation to uncover possible variation in in-use performance. There is some opinion among feldspar producers, and manufacturers of certain types of grinding mills, that a feldspar product closely sized below 325 mesh (44 microns) is superior in some applications.

(3) The fine quartz present appears amenable to beneficiation into pure silica product. A scavenger circuit might be required to attain greater purity than in Test No. 61 (Table 5).

(4) A mica fraction is present in the "fine" fraction, and apparently responds well to the USBM-type mica float procedure. It is probably about 7 to 8 percent of the total sample. Further research is needed, running a test series designed to float the mica selectively.

(5) An effective settling procedure for hydroclassification is not difficult to establish and operate. This is demonstrated by screen analyses cited, and by average particle size determinations.

(6) Use of a spar cleaner circuit seems useful in removing a fraction which is low in K_2O and high in CaO, Fe_2O_3 and quartz (Table 5).

Possible Future Research

Based on the findings of this preliminary work, the following areas could be researched in the future:

(1) Pulp density, duration, and additives: scrubbing cycle.

(2) Hydroclassification technique, using a cyclone to duplicate settling.

(3) Further investigation of conditioning variables: duration, pulp density, reagent level, pH, etc. to improve feldspar grade.

(4) Use of USBM-type mica float to obtain an acceptable mica product.

(5) Tests of the feldspar concentrate to determine its

useful application in ceramics and fillers.

(6) Further investigation of flotation techniques (cleaner floats, etc.) to aid beneficiation of mica, spar, and quartz products.

(7) Beneficiation of the "slimes" fraction to improve color and Fe_2O_3 analysis, or end-use research on this product as is.

(8) Assuming development of encouraging data, the operation of a pilot plant with filter cake as plant feed.

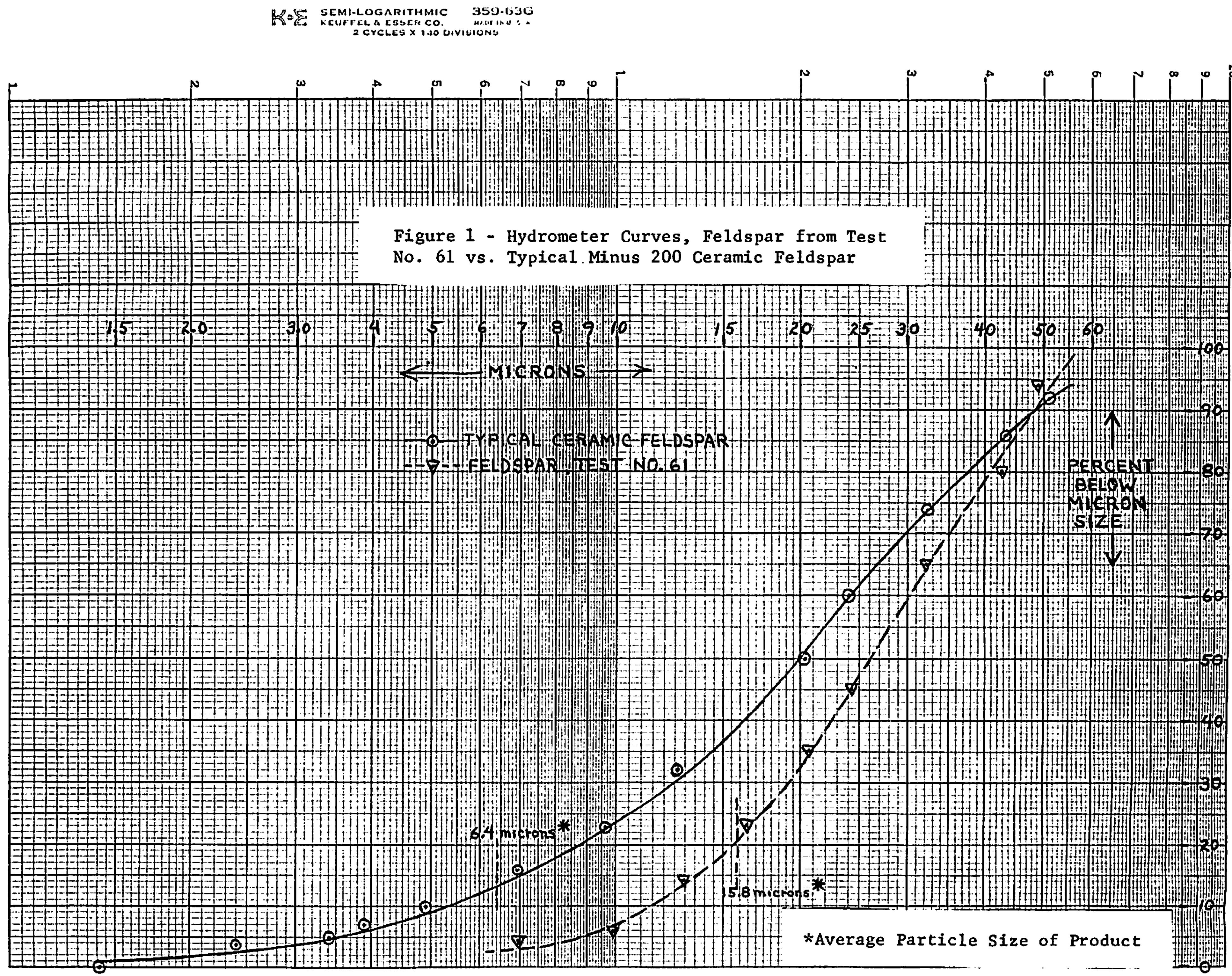

Figure 1 - Hydrometer Curves, Feldspar from Test No. 61 vs. Typical Minus 200 Ceramic Feldspar

Table 1

Comparative Screen Analysis, Sample #3300

vs. Long-Period Composite

Screen (Tyler)	Sample #3300		Composite	
+65	0.7		2.5%	
65-100	0.9		1.4%	
100-150	1.5		1.4%	
150-200	2.6		3.5%	
200-270	2.9		4.7%	
270-325	8.8		8.3%	
325-400	6.1	[Total-325	78.2	[Total-325]*
-400	76.5	=82.6%]	----	=78.2%]
Total	100.0%		100.0%	

*Only minus 325 fraction separated.

Table 2

Hydroclassified Products of Scrubbed Sample No. 3300
(Test No. 5)

	Weight %			
Tyler Screen	Coarse 6.8%	Intermediate 29.5%	Fine 38.7%	Slimes 25.0%
+20	----			
20-35	1.2			
35-48	2.9			
48-65	5.1			
65-100	9.8			
100-150	10.6	0.9		
150-200	15.7	3.4	0.2	
200-270	16.5	5.4	0.4	
270-325	24.1	26.6	0.9	
325-400	6.5	12.2	1.0	
-400	7.6	51.5	97.5	(Substantially 100%)
Total	100.0	100.0	100.0	100.0

Note: A material balance calculation yields a figure of 78.4% minus 400 mesh for the total Sample, compared to an actual screening figure of 76.5% (Table 1).

Table 3

Comparative Screen Analyses, "Fine" from Test No. 30 vs. "Fine" plus "Intermediate" from Test No. 5*

Tyler Screen	Test No. 5*	Test No. 30
+150	0.4	0.2
-150+200	1.6	2.2
-200+270	2.6	2.6
-270+325	12.0	13.5
-325+400	5.8	6.5
-400	77.6	75.0
Total	100.0	100.0
% of original sample	68.2	65.3

*Figures on Test No. 5 are materials balance calculations.

Table 4

Data on Significant Flotation Tests, "Fine" Fraction of Sample No. 3300

Test No.	Mica-Iron Float: Cond. Time (Mins.)	Mica-Iron Float: Cond. pH	Mica-Iron Float: M-70 (lbs/T)	Spar Float (lbs/T) Amine Acet.	% Mica-Iron F. P.	% Spar	% Spar Mids	% Qtz.	Fe_2O_3 Analyses: Spar	Fe_2O_3 Analyses: Mids	Fe_2O_3 Analyses: Qtz.	Fe_2O_3 Analyses: Spar & Qtz.
33	3	3.5	1.7	-	17.8	-	-	-	-	-	-	0.13
34	3	3.9	1.7	-	30.5	-	-	-	-	-	-	0.15
35	3	3.2	1.7	-	14.0	-	-	-	-	-	-	0.27
36	3	3.5	1.7	-	15.5	-	-	-	-	-	-	0.26
38	5	3.4	1.7	-	18.7	-	-	-	-	-	-	0.08
40	9	3.5	1.7	-	16.8	-	-	-	-	-	-	0.08
54	5	3.3	1.5	0.15	16.1	24.5	-	59.4	0.08	-	-	-
55	5	3.7	1.7	0.20	16.2	6.8	31.0	46.0	0.17	-	-	-
56	5	3.9	1.7	0.25	17.6	4.2	28.8	49.5	0.93	-	-	-
57	5	3.9	1.7	0.30	17.2	11.0	28.3	43.5	0.56	-	-	-
59	5	2.8	1.7	0.50	12.8	52.8	17.2	17.2	0.11	0.03	0.03	0.08[2]
61[1]	5	3.4	1.7	0.40	15.1	58.4	7.5	19.0	0.08	0.07	0.04	0.07[2]
62	5	3.7	2.0	0.35	15.6	51.0	12.8	20.6	0.07	0.08	0.04	0.07[2]

[1] See Table 5 for further data.
[2] Calculated.

Table 5

Physical and Chemical Characteristics, Products from Test No. 61

	"Coarse" Frac.	"Slimes"	"Fine" Fraction (65.3% of Total Sample) Mica-Iron F. P.	Spar	Spar Mids	Quartz
Percent of Classified Fraction	100	100	15.1	58.4	7.5	19.0
Percent of Total Sample No. 3300	4.4	30.3	9.8	38.2	4.9	12.4
Screen Analysis, Tyler: +20	-	-	-	-	-	-
20-35	4.7	-	-	-	-	-
35-48	8.5	-	-	-	-	-
48-65	16.4	-	-	-	-	-
65-100	27.1	-	-	Tr.	-	-
100-150	24.0	-	-	0.2	-	-
150-200	9.3	-	-	0.5	-	-
200-270	10.0	-	-	1.2	-	-
270-325	-	-	-	9.2	-	-
325-400	-	-	-	5.1	-	-
-400	-	100.0	-	83.8	-	-
Total	100.0	100.0	-	100.0	-	-
Avg. Part. Size (Microns)*	-	1.4	-	15.8	12.8	16.6
Chemical Analysis: % K_2O	-	3.90	-	5.26	1.49	0.35
% Na_2O	-	3.28	-	6.55	3.48	0.55
% CaO	-	-	-	1.28	0.80	Tr.
% Al_2O_3	-	-	-	20.80	10.60	1.70
% Fe_2O_3	-	2.16	-	0.075	0.065	0.035
% SiO_2	-	-	-	65.80	82.80	96.80
Ignition Loss, %	-	4.82	-	0.07	0.13	0.27

*Fisher Subsieve Sizer

Table 6

Comparison, Feldspar Product from Test No. 61 vs. Typical Sanitary-Grade Fine-Ground Feldspar

		Feldspar, Test No.61	Typical Sanitary-Grade Spar Product
Percent Minus 200 Mesh		99.3	99.3
Percent Minus 325 Mesh		88.9	93.2
Percent Minus 400 Mesh		83.8	----
Av. Part. Size (Fisher S. S. Sizer)		15.8 microns	6.4 microns
Chem. Analysis:	SiO_2	65.80	67.60
	Al_2O_3	20.80	19.05
	Fe_2O_3	0.075	0.045
	CaO	1.28	1.59
	K_2O	5.26	5.00
	Na_2O	6.55	6.40
	Ign. Loss	0.07	0.10