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Recovery of Feldspar and Glass Sand From Georgia Waste Granite Fines

By W. H. Eddy, E. W. Collins, and G. V. Sullivan
Tuscaloosa Metallurgy Research Center, Tuscaloosa, Ala.



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RECOVERY OF FELDSPAR AND GLASS SAND FROM GEORGIA WASTE GRANITE FINES

by

W. H. Eddy,¹ E. W. Collins,² and G. V. Sullivan³

ABSTRACT

The Bureau of Mines conducted laboratory batch- and small-scale continuous flotation processing tests on waste granite fines from Georgia, to determine the feasibility of recovering usable feldspar and quartz products by flotation.

Two flotation methods were utilized for the removal of biotite and other iron-bearing minerals: One was in an acid-cationic circuit, and the other in an alkaline-anionic-cationic circuit. Feldspar was recovered using an acid-cationic method. To produce commercial-grade feldspar and quartz products it was necessary to employ either two sequential froth flotation circuits or one froth flotation circuit supplemented by wet-magnetic separation techniques to remove iron contaminants.

Continuous flotation processing yielded a commercial-grade feldspar concentrate containing, in percent, 3.8 Na₂O, 5.4 K₂O, and 1.10 CaO, with recoveries of 77.8, 70.3, and 64.4, respectively. The feldspar tailing was high-quality glass sand containing 98.2 percent SiO₂ and 0.024 percent Fe₂O₃. About 42 percent of the quartz was recovered at glass sand specifications. An effective separation of potash feldspar from the bulk feldspar concentrate was achieved by batch flotation in a mineral separation cell.

INTRODUCTION

Feldspar deposits are widely scattered throughout the United States, but production is centered in four geographical locations: the Southeast, New England, the Rocky Mountains, and California.⁴ Reserves are conservatively estimated at 500 million long tons in deposits of the type currently exploited

¹Research technologist.

²Minerals engineer (now deceased).

³Supervisory metallurgist.

⁴Wells, J. R. Feldspar, Nepheline Syenite, and Aplite. BuMines Minerals Yearbook 1971, v. 1, 1973, pp. 499-507.

for their feldspathic content. North Carolina and California are the foremost producers; collectively they produce 65 percent of the domestic production.⁵

Most products in which feldspar is used also require silica, which has resulted in the increased use of feldspar-silica mixtures. The use of feldspathic sand increased from 132,000 tons in 1967 to about 230,000 tons in 1970 and 1971.⁶ Generally, commercial specifications for glass sand and feldspar, which vary according to use, require that Fe_2O_3 content be below 0.10 percent.

Results of previous research by the Bureau of Mines indicated that feldspar and quartz of acceptable grade⁷ could be separated from a Missouri granite. Other research involved the separation of mica, feldspar, and quartz from Minnesota granites, and feldspar and quartz from a waste granite fines from South Carolina.⁸

The granite fines is a waste product from quarrying and crushing operations. One quarry produces 180,000 tons of granite fines annually. (However, at present, all fine production is now sold for hot mix asphalt.) Feldspar and quartz make up the major part of the granite gneiss. Little attention has been given to the possibilities of obtaining feldspar and quartz from these granite fines. The Bureau of Mines initiated studies to determine the feasibility of utilizing these materials as a part of its primary mission of conservation of domestic minerals. Environmental aspects of the present method of fines disposition were also of primary importance in initiating the study. Results of the first phase of research on South Carolina granite fines has been reported.⁹ The present investigation is a continuation of these studies.

DESCRIPTION OF SAMPLE

The material used in this investigation was a waste granite fines product resulting from the screening of a fine-grained biotite granite gneiss mined by Consolidated Quarries Div., Georgia Marble Co., at Lithonia, Ga. The granite is crushed and then screened at 3 mesh. From the resulting minus 3-mesh material, a 6-ton sample was obtained for study. Representative samples were taken for chemical, petrographic, screen analysis, and laboratory batch flotation tests.

A chemical analysis of the ore is given in table 1 and petrographic analysis in table 2. Most of the iron present was contained in the biotite, and since removal of all the iron is a major consideration, for purposes of simplicity, this fraction will be referred to as biotite.

⁵Cooper, J. D., and J. R. Wells. Feldspar. Ch. in Mineral Facts and Problems. BuMines Bull. 650, 1970, pp. 977-988.

⁶Work cited in footnote 4, p. 500.

⁷Hill, T. E., Jr., H. Kenworthy, R. A. Ritchey, and J. A. Gerard. Separation of Feldspar, Quartz, and Mica From Granite. BuMines RI 7245, 1969, 25 pp.

⁸Eddy, W. H., E. W. Collins, J. S. Browning, and G. V. Sullivan. Recovery of Feldspar and Glass Sand From South Carolina Waste Granite Fines. BuMines RI 7651, 1972, 11 pp.

⁹Work cited in footnote 8.

TABLE 1. - Chemical analysis of waste granite fines

Constituent	Analysis, percent
Na ₂ O.....	3.3
K ₂ O.....	4.8
CaO.....	1.0
Al ₂ O ₃	14.1
SiO ₂	73.9
Fe ₂ O ₃	2.3
MgO.....	.2
Loss on ignition, 1 hour at 1,000° C...	.2
Other.....	.2
Total.....	100.0

TABLE 2. - Petrographic analysis of waste granite fines

Mineral	Percent
Muscovite.....	2
Biotite.....	9
Microcline.....	30
Plagioclase.....	26
Quartz.....	31
Other ¹	2
Total.....	100

¹Titanite, apatite, zircon, rutile, and epidote.

Petrographic examination showed that the feldspars and quartz were substantially free of locked gangue in the minus 35-mesh fractions. The major mineral constituents were quartz, plagioclase (oligoclase), microcline, biotite, and minor amounts of titanite, apatite, zircon, rutile, and epidote. To determine the composition of heavy minerals a sample of the material was separated in heavy liquid at a specific gravity of 3.30. Petrographic analysis of the heavy mineral fraction showed it to contain, in percent, 38 titanite, 40 opaque (principally magnetite, pyrite, and ilmenite), 10 zircon, 3 epidote, and minor quantities of staurolite, monazite, rutile, and biotite.

A screen analysis of the as-received material is shown in table 3.

TABLE 3. - Screen analysis of as-received waste granite fines

Screen size, mesh	Weight-percent	Screen size, mesh	Weight-percent
Plus 3.....	0.2	Minus 65 plus 100.....	6.2
Minus 3 plus 10.....	29.0	Minus 100 plus 150.....	5.2
Minus 10 plus 20.....	14.3	Minus 150 plus 200.....	4.7
Minus 20 plus 28.....	7.3	Minus 200 plus 270.....	2.8
Minus 28 plus 35.....	7.7	Minus 270 plus 400.....	2.1
Minus 35 plus 48.....	8.2	Minus 400.....	5.7
Minus 48 plus 65.....	6.6	Composite.....	100.0

FLOTATION STUDIES

Batch Testing

Laboratory batch flotation tests were undertaken to determine the optimum conditions for separating biotite, feldspar, and quartz.

Removal of Biotite in an Acid Circuit

A sample of the waste granite fines was stage ground in a pebble mill to pass a 35-mesh screen (mineral liberation size). The ground pulp was blunged with sodium hydroxide and the minus 400-mesh material was removed by screening. The pulp was conditioned with sulfuric acid, tallow amine acetate, kerosene, and methyl isobutyl carbinol. A biotite rougher product was floated. The rougher tailing was thickened to 40 percent solids and conditioned with hydrofluoric acid, tallow amine acetate, fuel oil, and pine oil. A rougher feldspar concentrate was then floated. A summary of results is shown in tables 4 and 5. About 76 percent of the iron-bearing minerals present in the ore was removed in the biotite flotation circuit.

Petroleum sulfonates were also studied as collectors for iron-bearing minerals in acid circuits. The bulk of the iron-bearing minerals were responsive to such treatment, but the results were not as good as those obtained with amines.

TABLE 4. - Removal of biotite in an acid circuit

Product	Weight-percent	Analysis, percent				Distribution, ¹ percent			
		Na ₂ O	K ₂ O	CaO	Fe ₂ O ₃	Na ₂ O	K ₂ O	CaO	Fe ₂ O ₃
Oversize.....	0.2	-	-	-	-	-	-	-	-
Slimes.....	20.6	-	-	-	-	-	-	-	-
Biotite rougher product...	7.6	-	-	-	-	-	-	-	-
Feldspar concentrate.....	47.5	5.0	7.2	1.3	² 1.10	72.0	71.2	61.8	22.7
Feldspar tailing.....	24.1	0.1	0.1	0.2	0.08	0.7	0.5	4.8	0.8
Feldspar composite...	71.6	2.4	3.4	0.7	0.54	72.7	71.7	66.6	23.5
Total ³	100.0	3.3	4.8	1.0	2.3	-	-	-	-

¹Distribution calculated from the assay of the head sample.

²Fe₂O₃ reduced to 0.09 percent by wet magnetic separation.

³Head sample assay.

TABLE 5. - Reagent schedule for biotite removal and feldspar flotation

Operating conditions	Blunge	Biotite conditioner	Rougher cell	Feldspar conditioners		Rougher cell
				No. 1	No. 2	
Reagents, pounds per ton of ore:						
Sodium hydroxide.....	1.5	-	-	-	-	-
Sulfuric acid.....	-	2.0	-	-	-	-
Tallow amine acetate.....	-	0.25	-	-	1.0	-
Kerosene.....	-	0.48	-	-	-	-
Methyl isobutyl carbinol....	-	0.12	-	-	-	-
Hydrofluoric acid.....	-	-	-	1.0	-	-
Fuel oil No. 5.....	-	-	-	-	0.48	-
Pine oil.....	-	-	-	-	0.32	-
Conditioning time....minutes..	5	1-1/2	2	5	2	1-1/2
Temperature.....° C..	-	27	-	-	31	-
Pulp pH.....	-	2.3	-	-	3.3	-

Effect of pH on Biotite Removal in Acid Circuits

A series of flotation tests was made to study the effect of pH on biotite flotation. The waste granite fines material was prepared as previously described. The pulp was conditioned at 40 percent solids in a Minerals Separation-type cell¹⁰ with various quantities of sulfuric acid to vary the pH and depress the feldspar and quartz, and with 0.25 pound per ton of tallow amine acetate and 0.48 pound per ton of kerosene as collectors, and 0.12 pound per ton of methyl isobutyl carbinol as a frother to float the biotite. A biotite product was floated and discarded. The results are shown in table 6.

TABLE 6. - Effect of pH on biotite removal in acid circuits

Sulfuric acid, pounds per ton of ore	Sample pH	Biotite product		
		Weight-percent	Fe ₂ O ₃ analysis, percent	Fe ₂ O ₃ distribution, percent
0.5	4.7	17.7	7.9	60.8
1.0	2.7	12.0	10.8	56.3
1.5	2.6	9.3	16.9	68.3
2.0	2.3	8.7	17.1	64.7
2.5	2.2	7.8	19.2	65.1
3.0	2.1	8.4	18.5	67.6
3.5	2.1	8.5	19.3	71.3
6.5	1.8	8.2	18.9	67.4
9.0	1.8	8.0	18.7	65.0
-	-	-	¹ 2.3	-

¹Based on head sample.

¹⁰Reference to specific company or product names is made for identification only and does not imply endorsement by the Bureau of Mines.

The results indicate that maximum iron removal occurred at pH of 2.1. The biotite concentrate contained 19.3 percent Fe_2O_3 , representing 71.3 percent of the total iron.

Removal of Biotite Sequentially in Alkaline and Acid Circuits

A sample of the waste granite fines was stage ground to minus 35 mesh with sodium hydroxide in a pebble mill. The pulp was washed free of sodium hydroxide, as the calculated minus 12-micrometer material was removed by decantation. The pulp was conditioned with slow agitation at 70 percent solids with sodium carbonate to regulate the alkalinity, calcium magnesium lignin sulfonate to disperse the pulp and depress the feldspar and quartz, and tall oil and tallow amine acetate to collect the finer sized biotite. Following this, a primary biotite rougher product was floated. The tailing was thickened to 40 percent solids and conditioned with sulfuric acid to regulate the acidity and depress the feldspar and quartz, and with a mixture of two petroleum sulfonates to collect the coarser sized biotite. A secondary biotite rougher product was floated. The biotite rougher tailing contained, in percent, 3.6 Na_2O , 4.5 K_2O , 0.56 CaO , 12.9 Al_2O_3 , 77.1 SiO_2 , and 0.09 Fe_2O_3 , with a distribution, in percent, of 85.2 Na_2O , 73.2 K_2O , 43.7 CaO , 71.4 Al_2O_3 , 81.5 SiO_2 , and 3.5 Fe_2O_3 . About 38 percent of the SiO_2 was free quartz. The product was feldspathic sand quality. A summation of results is shown in tables 7 and 8.

TABLE 7. - Removal of biotite sequentially in alkaline and acid circuits

Product	Weight-percent	Na_2O	K_2O	CaO	Al_2O_3	SiO_2	Fe_2O_3
Analysis, percent							
Grind and thickener slimes.....	6.5	-	-	-	-	-	4.51
Biotite primary rougher product..	10.8	-	-	-	-	-	8.90
Biotite secondary rougher product	4.6	-	-	-	-	-	14.50
Tailings (feldspathic sand).....	78.1	3.6	4.5	0.56	12.9	77.1	0.09
Total.....	100.0	¹ 3.3	¹ 4.8	¹ 1.00	¹ 14.1	¹ 73.9	² 1.99
Distribution, ³ percent							
Grind and thickener slimes.....	-	-	-	-	-	-	14.7
Biotite primary rougher product..	-	-	-	-	-	-	48.3
Biotite secondary rougher product	-	-	-	-	-	-	33.5
Tailings (feldspathic sand).....	-	85.2	73.2	43.7	71.5	81.5	3.5
Total.....	-	-	-	-	-	-	100.0

¹Head sample assay.

²Calculated head sample. Head sample assay listed in table 1.

³Distribution calculated from the assay of the head sample.

TABLE 8. - Reagent schedule for biotite removal
in alkaline and acid circuits

Operating conditions	Scrub	Alkaline circuit conditioner	Acid circuit conditioner
Reagents, pounds per ton of ore:			
Sodium hydroxide.....	1.5	-	-
Sodium carbonate.....	-	1.0	-
Calcium magnesium lignin sulfonate...	-	1.0	-
Tall oil.....	-	0.32	-
Tallow amine acetate.....	-	0.10	-
Sulfuric acid.....	-	-	0.6
Petrofloat 562 ¹	-	-	1.6
MORCO No. 62 ¹	-	-	1.4
Time.....minutes..	10	5	5
Temperature.....° C..	-	24	25
Pulp pH.....	-	8.9	2.1

¹Water-soluble petroleum sulfonates.

Selective Recovery of Potash Feldspar

A sample of the waste granite fines was ground with 1.5 pounds per ton sodium hydroxide to minus 35 mesh, the minus 400-mesh material was removed and the pulp was thickened to 40 percent solids and then conditioned with sulfuric acid, coco amine hydrochloride, kerosene, and methyl isobutyl carbinol. A rougher biotite product was floated. The biotite tailing was thickened to 40 percent solids and conditioned with hydrofluoric acid, tallow amine acetate, fuel oil, and pine oil. A bulk feldspar concentrate was floated, followed by blunging with sodium hydroxide and conditioned with^{1,1} sodium metasilicate, tallow amine acetate, kerosene, and pine oil. A rougher potash feldspar was floated and cleaned twice. The cleaner concentrate was potash feldspar, while the middlings were combined with the cleaner tailings to produce a mixed feldspar concentrate. The results are shown in tables 9 and 10.

TABLE 9. - Selective recovery of potash feldspar

Product	Weight-percent	Analysis, percent			Distribution, percent		
		Na ₂ O	K ₂ O	CaO	Na ₂ O	K ₂ O	CaO
Potash feldspar concentrate.	19.0	3.40	9.68	0.98	19.0	38.8	18.6
Mixed feldspar concentrate..	28.5	5.85	5.13	1.60	49.0	30.5	45.6
Total ¹	-	3.3	4.8	1.00	-	-	-

¹Head sample assay, as given in table 1.

^{1,1}Hall, D. J., Jr. (assigned to International Minerals & Chemical Corp., New York). Process for Beneficiating Ores. U.S. Pat. 3,113,922, Dec. 10, 1963.

TABLE 10. - Reagent schedule for selective recovery of potash feldspar

Operating conditions	Blunge	Potash feldspar conditioner	
		No. 1	No. 2
Reagents, pounds per ton of ore:			
Sodium hydroxide.....	2.0	-	-
Kerosene.....	-	-	0.16
Tallow amine acetate.....	-	-	0.50
Pine oil.....	-	-	0.16
Sodium metasilicate.....	-	3.5	-
Time.....minutes..	-	1	1
Temperature.....° C..	-	-	29
Pulp pH.....	-	-	10.9

An effective separation of potash feldspar from soda feldspar was achieved.

The product listed in table 9 as potash feldspar concentrate, containing 9.68 percent K_2O , approached the specifications for potash feldspar (10.0 percent K_2O). The ratio of $K_2O/Na_2O + CaO$ in the potash feldspar concentrate was 2.21, while the ratio of $K_2O/Na_2O + CaO$ in the feed was 1.09. A number of attempts were made during continuous processing to make a high-grade potash feldspar product, but the system was not steady enough.

Continuous Testing

Based on results of laboratory batch flotation tests, a continuous processing unit with a capacity of about 150 pounds of dry feed per hour was assembled. The process included grinding, classification, conditioning, flotation, and wet-magnetic separation as shown in figure 1.

The waste granite fines (minus 3 mesh), which were stored in a bin, were fed to a rodmill by means of a constant-weight feeder. Sodium hydroxide was added to the rodmill to disperse the slimes. The rodmill was operated in closed circuit with a trommel screen to grind the ore to minus 35 mesh. The screen undersize material was pumped to a hydroseparator for thickening and removal of the minus 400-mesh slimes. The classifier sand was pumped to a bowl-rake classifier for an additional cleaning to assure removal of the minus 400-mesh slimes. The resultant sand was diluted to 40 percent solids and flowed by gravity to a conditioner, where sulfuric acid, tallow amine acetate, kerosene, and methyl isobutyl carbinol were blended with the pulp at slow speed. A 7-minute retention time was used. A biotite rougher product was then floated as a waste product. The biotite tailing was allowed to flow by gravity to a high-intensity wet-magnetic separator for the removal of additional magnetic material. The nonmagnetic product was then thickened in a rake classifier prior to feldspar flotation. The classifier overflow was used as makeup water in the feldspar rougher flotation circuit. The classifier sand was diluted to 40 percent solids and conditioned for 8 minutes at slow speed with hydrofluoric acid, tallow amine acetate, and fuel oil. Pine oil was added to the pulp as it entered a bank of three flotation cells, where a feldspar concentrate was floated.

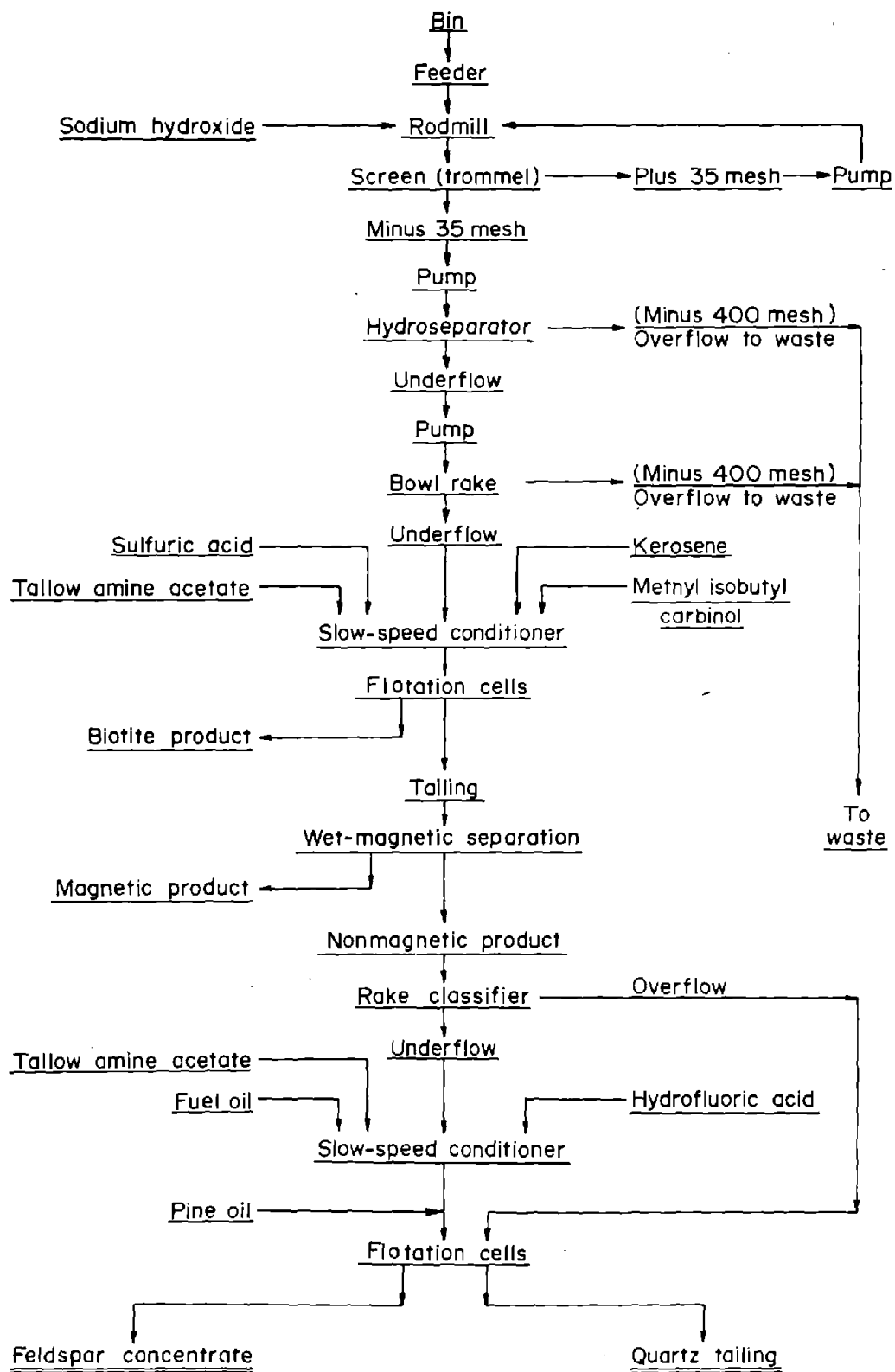


FIGURE 1. - Continuous flotation for recovery of feldspar and quartz from Georgia waste granite fines.

All the conditioning was done with a minimum of agitation and residence time to reduce degradation of the ore. Results of continuous testing are shown in tables 11 and 12.

TABLE 11. - Continuous flotation for recovery of feldspar and quartz

Product	Weight-percent	Na ₂ O	K ₂ O	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃
		Analysis, percent					
Bowl rake and hydroseparator overflow.....	9.0	3.9	5.6	1.20	13.6	67.3	3.03
Biotite product.....	9.2	2.1	7.8	1.93	17.7	45.3	17.40
Biotite tailing magnetic fraction.....	4.7	2.9	4.8	2.07	12.0	72.7	4.40
Feldspar concentrate.....	64.1	3.8	5.4	1.10	16.1	71.3	0.092
Feldspar tailing.....	13.0	0.1	0.1	0.05	1.3	98.2	0.024
Feldspar composite.....	77.1	3.2	4.5	0.92	13.6	75.8	0.08
Total ¹	100.0	3.1	4.9	1.09	13.9	72.1	2.14
		Distribution, percent					
Bowl rake and hydroseparator overflow.....	-	11.2	10.2	9.9	8.8	8.4	12.7
Biotite product.....	-	6.2	14.6	16.2	11.7	5.8	74.7
Biotite tailing magnetic fraction.....	-	4.4	4.6	8.9	4.1	4.7	9.7
Feldspar concentrate.....	-	77.8	70.3	64.4	74.2	63.4	2.8
Feldspar tailing.....	-	0.4	0.3	0.6	1.2	17.7	0.1
Feldspar composite.....	-	78.2	70.6	65.0	75.4	81.1	3.0
Total.....	-	100.0	100.0	100.0	100.0	100.0	100.0

¹Calculated head sample. Head sample assay listed in table 1.

TABLE 12. - Reagent schedule for continuous flotation for recovery of feldspar and quartz

Operating conditions	Grind	Biotite conditioner	Feldspar conditioner
Reagents, pounds per ton of ore:			
Sodium hydroxide.....	1.5	-	-
Sulfuric acid.....	-	3.00	-
Tallow amine acetate.....	-	0.30	1.90
Kerosene.....	-	0.48	-
Methyl isobutyl carbinol.....	-	0.12	2.25
Hydrofluoric acid.....	-	-	0.48
Fuel oil.....	-	-	0.32
Pine oil.....	-	-	-
Temperature.....° C..	-	22	23
Time.....minutes..	-	7	8
Pulp pH.....	-	2.1	3.5

The feldspar concentrate contained, in percent, 3.80 Na₂O, 5.40 K₂O, and 1.10 CaO, with a total alkaline content of 10.30 percent, and a recovery, in

percent, of 77.8 Na₂O, 70.3 K₂O, and 64.4 CaO. The feldspar tailing (quartz concentrate) was a high-quality glass sand product containing, in percent, 98.2 SiO₂ and 0.024 Fe₂O₃, at a recovery of about 42 percent.

CONCLUSIONS

1. The laboratory batch and continuous processing unit tests demonstrated the feasibility of recovering high-grade feldspar and quartz products from Georgia waste granite fines; biotite removed was accomplished by either two sequential froth flotation circuits or one flotation circuit supplemented by wet-magnetic separation.

2. A high-grade feldspathic sand was made by removal of fine-size biotite in an alkaline circuit followed by removal of coarse-size biotite in an acid circuit.

3. The optimum pH in an acid flotation circuit for removal of biotite was 2.1. A biotite product containing 19.3 percent Fe₂O₃ was removed, representing 71.3 of the total iron in the ore.

4. Removal of the primary and secondary slimes at 400 mesh was sufficient to obtain good froth flotation.

