

A Method for Concentration of North Carolina Spodumene Ores

by Mason K. Banks, William T. McDaniel, and Philip N. Sales

A process has been developed which produces spodumene concentrates assaying 6.0 pct Li_2O and 0.45 pct Fe_2O_3 , with 70 to 75 pct recovery of spodumene. Two flotation separations are required: the simultaneous removal of mica, feldspar, and quartz as a froth product using a cationic collector, and the subsequent removal of iron-bearing minerals as a froth product while spodumene is depressed as a tailing.

SINCE the close of World War II there have been numerous developments resulting in the increased use of lithium by industry. Notable among these has been the development of an industrial lubricant, based on lithium stearate, which is fluid at low temperatures, stable at high temperature, and insoluble in water. A new bleaching agent, lithium hypochlorite, has also been developed recently.¹ Other well-known uses are listed below:

1—Lithium oxide in ceramics reduces firing time, lowers melting temperatures, lowers thermal expansion, raises refractive index, and improves chemical resistivity. 2—Lithium carbonate and lithium strontium nitrate are used in pyrotechnics for their brilliant red color.² 3—Lithium chlorides and fluorides are used as fluxes for welding aluminum and magnesium.² 4—The affinity of lithium for nitrogen is the basis for a process used in purifying helium.² 5—Lithium chlorides and bromides are used in air conditioning for their powers to absorb organic amines, ammonia, and smoke.¹ 6—Lithium hydride has been used as a source of hydrogen to inflate life rafts and balloons used by the Air Forces and Navy.¹ 7—Lithium metal and lithium hydride are useful in the atomic energy program in connection with the production of the isotopes of hydrogen.³

During the 1942-45 period of World War II, Solvay Process Co. operated a flotation plant with a daily capacity of 300 tons of ore for the production of spodumene concentrate near Kings Mountain, North Carolina. The operation was shut down upon reduction of wartime demand for lithium chemicals, and

M. K. BANKS, Chief Engineer, W. T. McDANIEL, Member AIME, Ore Dressing Engineer, and P. N. SALES, Chemist, are with the North Carolina State College of Agriculture and Engineering Minerals Research Laboratory.

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until recently there has been no production from the Carolinas.

In April 1950 an investigation of the problem of spodumene flotation from North Carolina ores was begun at North Carolina State College Minerals Research Laboratory at Asheville, N. C. This laboratory, operating under the combined auspices of North Carolina State College, North Carolina Department of Conservation and Development, and the Tennessee Valley Authority, undertook the spodumene project at the request of Foote Mineral Co. of Philadelphia. The work, involving a period of 15 months of intensive batch and pilot plant investigation, was completed in June 1951 and resulted in development of a process for concentration of spodumene by flotation which has been hereto unrecorded in the literature. A similar approach has been used before on iron, ilmenite, and other minerals, but the literature shows no record of its having been applied to spodumene. Parts of the process are now being used on a commercial scale at the Foote Mineral Co. plant near Kings Mountain, N. C., which has been in production since July 1951. It is the purpose of this paper to describe some of the results of the investigation.

The Orebodies

Spodumene-bearing pegmatites occur in a narrow belt 24.5 miles long and 1.8 miles wide extending southwestward from Lincolnton to Grover, N. C. According to statements judged to be conservative, the tin-spodumene belt of the Carolinas contains an estimated 4,300,000 tons of pegmatite ore averaging 15 pct spodumene. This amounts to "a reserve of at least 650,000 tons of spodumene (more than 20,000 tons of metallic lithium) to a depth of 100 ft." This figure includes only bodies having an average thickness of 35 ft and length of 550 ft. There are hundreds of pegmatites in the belt, many of which are less than 10 ft wide. The largest pegmatite in the area has a maximum thickness of 395 ft and maximum length of 3250 ft.⁴

These larger pegmatites are composed principally

of albite and microcline feldspars, albite predominating, quartz, spodumene, and muscovite. There are many accessory minerals, but the important ones are cassiterite and beryl, and possibly columbite-tantalite. Approximate composition of ores from the belt which were used in this investigation is shown in Table I.

Table I shows that quartz and muscovite content remain fairly constant in all the ores, while feldspar content increases when spodumene content decreases, and vice versa. This is interesting because feldspar is potentially an important byproduct.

The Spodumene

According to Boyd,⁵ the following analysis is typical of the Carolina spodumene:

SiO ₂	Al ₂ O ₃	Li ₂ O	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	Ignition Loss
63.84	28.61	7.26	0.19	0.04	Trace	Nil	Nil	0.16

Spectrographic analysis shows the presence of minor amounts of MnO, TiO₂, SnO₂, Ga₂O₃, Na₂O, and K₂O in the spodumene. One sample contained Rb₂O. Petrographic and X-ray studies indicated that these impurities are probably isomorphous replacements in the crystal structure of the spodumene.⁶

The molecular dispersion of the iron within the spodumene crystal has been substantiated by acid-leaching and magnetic separation tests in which the Fe₂O₃ content of spodumene crystals remained from 0.15 to 0.40 pct after magnetic separation and acid leaching.⁷

The iron content of the spodumene crystals is usually reflected in the color of the crystals, the colorless to white variety containing from 0.08 to 0.20 pct Fe₂O₃, the buff or cream variety containing 0.20 to 0.40 pct Fe₂O₃ and the yellow or greenish-yellow variety containing up to 0.65 pct Fe₂O₃. The darker the color the higher the iron content of the spodumene.

The amount of iron dispersed in the spodumene crystals is a significant factor, since it affects the use to which the concentrates may be put. For use in ceramics the spodumene concentrate should contain less than 0.5 pct Fe₂O₃, but for use in extraction of lithia for chemical purposes the presence of iron presents no special problem. None of the Carolina belt samples tested contained spodumene with low enough isomorphous iron content to yield a product with less than 0.2 pct Fe₂O₃. The average iron content of spodumene concentrates prepared from these ores was 0.40 to 0.50 pct.

The Carolina spodumene is somewhat variable in Li₂O content, but apparently averages approximately 7.0 pct or slightly in excess of this figure, although the textbook gives 8.03 pct theoretical Li₂O. Some variation is caused by weathering, for it has been established that Li₂O is lost by leaching when the rock undergoes weathering, just as the Na₂O and K₂O are lost in kaolinization of feldspar.

Review of Concentration Methods

The literature reveals the following methods used for production of spodumene concentrates: 1—The spodumene of the Black Hills of South Dakota was present in such large crystals that it was easily concentrated by selective mining and hand sorting. 2—The Bureau of Mines developed a method by which alpha spodumene was decrepitated at a temperature between 1000° and 1100°C, changing its form to beta spodumene, a soft pulverulent material, which

could be recovered from the harder gangue minerals by differential grinding following the decrepitation.⁶ 3—Froth flotation of spodumene using anionic collectors, or fatty acids, in an alkaline circuit was worked out by the Bureau of Mines and enlarged upon by Norman and Gieseke.⁷ This method was utilized by Solvay Process Co. at Kings Mountain, N. C., during its wartime operation. 4—Froth flotation with sulphonated oils in an acid circuit was also shown to be feasible, as well as a method of producing a feldspar-spodumene mixture by using cationic collectors in an HF circuit.⁷ 5—A mill at Keystone, S. D., operated by Lithium Corporation of America has been utilizing the heavy media method, treating 15 tons of -1½ in. ore per hr and producing a concentrate of 4 to 5 pct Li₂O.⁸

All the above methods have their advantages and disadvantages. The method described on the following pages is presented merely as a different approach and is acknowledged also to have both advantages and disadvantages.

The Flotation Process

The final process developed for spodumene concentration of North Carolina ores revolves around two primary flotation separations: 1—a process whereby mica, feldspar, and quartz are simultaneously removed as a froth product while spodumene and iron-bearing minerals are depressed as a tailing, and 2—the subsequent removal of iron-bearing minerals as a froth product while spodumene is depressed as a tailing. The removal of silicate gangue minerals in Step 1 is accomplished in an alkaline circuit, with a long-chain amine used as collector, and a colloidal

Table I. Ores Used in Investigation of Spodumene Flotation

Ore, Lab. No.	Sampled by	Spodumene, Pet	Feldspar, Pet	Quartz, Pet	Mica, Pet	Others, Pet**
808	Laboratory staff	38	30	26	3	2
837-A	Footo Mineral Co.*	36	33	26	3	2
971	Consolidated Feldspar Corp.	23	41	27	5	4
943	Footo Mineral Co.	19	45	25	4	7
969	Footo Mineral Co.	22	42	25	4	6
985	Footo Mineral Co.	13	56	22	4	5

* The Footo samples contained 0.03 to 0.05 pct Sn, or approximately 1.0 lb Sn per ton of ore. These came from the old Solvay quarry about 1 mile SW of Kings Mountain.

** Includes hornblende gneiss and sericite schist particles from wall rock and inclusions, as well as beryl, cassiterite, and numbers of accessory pegmatite minerals present in trace quantities.

depressant, such as starch or dextrine, used as selective depressant for the spodumene and for the iron minerals. The iron minerals are removed in an acid circuit, with sodium resinate used as collector and hydrofluoric acid as controller for prevention of spodumene flotation. Suitable frothers are used in both floats.

The gangue froth product containing mica, feldspar, and quartz may be split into its component parts for recovery of a feldspar byproduct. Since approximately one-third of the feed is generally recoverable as a feldspar concentrate, it was felt that efforts to recover it should be made. The general procedure for feldspar recovery would be to dewater the gangue froth product and then treat it in the conventional manner for feldspar ores, i.e., removal of mica with sulphuric acid and amine, washing by classification, then flotation of other iron-bearing silicates with sulphonated oil or fatty acid collectors, and finally flotation of feldspar from quartz with the conventional hydrofluoric acid-amine process.

Table II. Batch Test Results

Spodumene									
Lab. No. 808 Head Sample: 38 pct spodumene, 0.24 pct Fe ₂ O ₃ in spodumene crystals.									
Product	Wt, Pet	Pct	Units	Dist.	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	(1) Li ₂ O	(2) Li ₂ O
Spodumene conc.	29.4	91	26.8	70.8	0.26			6.55	
Iron froth product	3.2	62	2.0	5.3				4.85	
Mica froth product	4.2	4	0.2	0.5				0.45	
Feldspar froth product	21.0	14	2.9	7.7	0.07		67.0	1.25	
Quartz tailing	29.8	8	2.4	6.3	0.04		93.3	0.75	
Slime and oversize	12.5	29*	3.6*	9.5*					
Total	100.1		37.9	100.1					
Spodumene									
Lab. No. 837-A Head sample: 36 pct spodumene, 0.43 pct Fe ₂ O ₃ in spodumene crystals.									
Product	Wt, Pet	Pct	Units	Dist.	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	(1) Li ₂ O	(2) Li ₂ O
Spodumene conc.	33.8	89	30.1	83.6	0.44			6.50	6.44
Iron froth product	1.9	50	1.0	2.8	8.29			4.05	4.50
Mica froth product	1.5	3	0.1	0.3				0.35	0.52
Feldspar froth product	27.5	6	2.2	6.1	0.09	19.9	68.4	0.70	0.68
Quartz tailing	20.9	1	0.2	0.6	0.02		98.0	0.10	0.11
Slime oversize	14.4	18*	2.4*	6.7*					
Total	100.0		36.0	100.1					
Spodumene									
Lab. No. 943 Head sample: 19 pct spodumene.									
Product	Wt, Pet	Pct	Units	Dist.	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	(1) Li ₂ O	(2) Li ₂ O
Spodumene conc.	14.3	82	11.7	63	0.86			6.10	6.40
Iron froth product	2.6	55	1.4	7				4.35	4.00
Mica froth product	6.0	5	0.3	2				0.50	1.19
Feldspar froth product	37.9	7	2.6	14	0.26	20.6	66.0	0.70	1.40
Quartz tailing	20.1	1	0.2	1	0.14		94.1	0.10	0.36
Slime oversize	19.1	13*	2.4*	13*					
Total	100.0		18.6	100					
Spodumene									
Lab. No. 971 Head sample: 23 pct spodumene, 0.47 pct Fe ₂ O ₃ in spodumene crystals.									
Product	Wt, Pet	Pct	Units	Dist.	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	(1) Li ₂ O	(2) Li ₂ O
Spodumene conc.	22.8	84	19.2	83.5	0.56			6.25	
Iron froth product	1.0	94	0.9	3.9				6.70	
Mica froth product	4.4	1	0.0	0.0				0.10	
Feldspar froth product	36.6	4	1.5	6.5	0.12	20.4	68.1	0.40	
Quartz tailing	20.8	1	0.2	0.9	0.05		96.5	0.10	
Slime and oversize	14.3	8*	1.2*	5.2					
Total	99.9		23.0	100.0					

(1) By using curve showing Li₂O vs percent sinks in acetylene tetrabromide.

(2) By actual chemical analysis.

* Calculated using head sample analysis.

Distribution of spodumene based on percentage of sinks in acetylene tetrabromide.

Li₂O analyses by Foote Mineral Co.

The final process which breaks the ore into its component parts is described generally as follows:

1—Grinding. The ore as received was ground in a batch rod mill at 25 pct solids to pass a 28-mesh screen. The +28 mesh fraction, consisting almost entirely of mica, was removed. The -28 mesh fraction was deslimed by decanting over a 325-mesh screen. Approximately 0.5 lb caustic per ton of feed was used in the mill to keep the pulp dispersed for desliming. The oversize product consisted of approximately 1.0 pct weight of the feed, while slime loss came to 12 to 18 pct weight of the feed. Separations were obtained with feed as coarse as 20 mesh, and the general range of 20 to 28 mesh was satisfactory. The feed should be sized by screening or classification, with oversize returning to the mill in practice. The rod mill is preferred to the ball mill, because it produces a coarser, more granular feed with much closer size distribution. It is believed that ball milling would produce excessive slimes and a long particle size range, both of which have been shown to be undesirable in this process.

2—Rougher Gangue Flotation. The deslimed -28 mesh +325 mesh pulp was placed in a Denver 2000-g laboratory flotation machine and brought to 20 to 25 pct solids. The following reagents, calculated in pounds per ton of dry mill feed, were added: 1.0 lb per ton of lime, 0.5 of dextrine, 0.4 to 0.7 of Armac T, and 0.4 of B-23. The pulp was conditioned for 1 min, then floated for 4 min, with the quartz, feldspar, and mica being simultaneously removed as a froth product. The spodumene and iron-bearing

minerals were selectively depressed and removed as a tailing. Most of the froth was removed in 2 min. The float is heavy and fast if sufficient frother is used. The rougher is more selective if little or no frother is used, but requires more collector (Armac T) and an excessive float time. Lime and dextrine are more selective and cheaper than caustic and dextrine, although either may be used. If dextrine is not available, causticized-starch may be used, although dextrine is preferable. An excess of starch or dextrine does not break down the separation, but merely causes an increase in collector required. Approximately 0.5 lb per ton of dextrine or causticized-starch is generally sufficient to depress the spodumene. An excess of caustic or lime can cause complete breakdown of the separation as the amine collector is unstable when the pH is above 11.5.

3—Cleaner Gangue Flotation. The rougher gangue froth was next placed in the cell at 15 to 20 pct solids with 0.5 lb per ton of lime, 0.5 of dextrine, and 0.2 of B-23. It was conditioned 15 sec, then floated 4 min. This is virtually a repetition of the rougher, for the purpose of dropping out fine spodumene particles which were mechanically entrained in the heavy rougher froth. It requires no additional collector. If sufficient frother is used, the cleaner tailing or middling is a high-grade spodumene concentrate and may be combined with the rougher tailing.

4—Iron Mineral Flotation. The combined rougher tailing and cleaner tailing, consisting of a high-grade spodumene concentrate with substantially all the

iron-bearing minerals present, was then washed by decantation, thickened to 65 to 75 pct solids, and conditioned for 10 min with 0.35 lb per ton of HF (100 pct), 2.0 of sodium resinate, and 0.1 of B-24. The pulp was transferred to the cell and floated 4 min at 25 pct solids, with iron-bearing minerals being removed as a froth product, and relatively iron-free spodumene, except for molecularly contained iron discharged as a tailing. The tailing is the finished spodumene concentrate, which may be thickened, filtered, and dried in conventional manner. The sodium resinate collector was chosen over sulphonated oils as iron-mineral collector because it tends to concentrate the spodumene by floating mica and feldspar impurities along with the iron minerals, whereas the sulphonated oils tend to float pure spodumene along with the iron minerals. It is most important to maintain pulp density in the conditioner as high as possible before flotation of iron minerals.

5—Mica Flotation. The cleaner gangue froth was next conditioned for 3 min with 1.0 lb per ton of H_2SO_4 , 0.1 of B-23, and 0.05 of Armac T at 35 pct solids. Mica was then removed as a froth product.

6—Feldspar Flotation. The quartz-feldspar tailing from the mica float was washed by decantation, then conditioned 6 min at 50 pct solids with 1.5 lb per ton of HF (100 pct). It was then placed in the cell and diluted to 25 pct solids; 0.05 to 0.20 lb per ton of Armac T and 0.3 of B-23 were added before flotation. Feldspar was removed as a froth product and quartz was discharged as a tailing.

Results on batch testing several ores according to this outlined process are shown in Table II. Fig. 1 is a flowsheet of the process.

Discussion of Table II

Test results on four separate ores are shown in Table II to illustrate the amenability of various ore types and grades to separation by this method. Lithia content is given in two of the test summaries to show the comparison between actual chemical analysis for lithia and lithia content based on distribution of spodumene. It should be observed that lithia minerals must be considered on a dual basis: first, the mineral, which is all the mill operator can recover, and second, the lithia content, which is all-important to industry. Recoverable spodumene crystals in some instances may not contain more than 3.0 to 5.0 pct Li_2O owing to loss by leaching action of weathering, replacement of Li_2O by other molecules, etc. Other points which should be noted in explaining the seeming discrepancies between spodumene content and lithia analysis are: 1—The mica in the pegmatites contains a small amount of Li_2O . This throws any attempt to check spodumene distribution with Li_2O distribution slightly off. 2—The Li_2O content of the spodumene itself is variable. 3—Accuracy of Li_2O analysis is subject to serious error because of its complexity.

For these reasons, results were evaluated in terms of spodumene distribution rather than lithia distribution. Actual lithia analyses were made at intervals for check purposes and these are shown where available.

It will be noted that the iron content of the spodumene concentrates is usually slightly above that of the picked spodumene crystals from a given sample. The iron content of the crystals is part of the molecule and cannot be removed by physical means.

Ores 808 and 837-A were higher grade than average and in fresh, clean condition. No trouble was encountered in making very good lithia grades and

recoveries on this type of ore. Feldspar concentrates were also of good quality on these two ores.

Ore 971 was a medium-weathered, high-iron content spodumene ore, but was still definitely amenable to this process, as shown by the results.

Ore 973 was badly weathered and contained considerable kaolinized material and hornblende. This resulted in high-iron feldspar concentrates and high-iron spodumene concentrates. This ore severely tested the process, but spodumene concentrates containing 6.10 pct Li_2O minimum were prepared from it with a recovery of 63 pct. One of the chief conclusions drawn from handling ores like this was that an acceptable grade of concentrate could be made from badly weathered ores, but recovery would suffer. Hornblende was outstanding as the most difficult contaminant to cope with. It consistently reported with the spodumene concentrates when present in the ore, and was not easily separated therefrom. The chief loss of spodumene values occurred in the slimes, and the next highest loss in the feldspar concentrate. The spodumene recoveries shown compare favorably with those obtained in feldspar flotation, with which this type of processing is most closely comparable.

Pretreatment of Weathered Ores

It was found necessary to employ light attrition scrubbing on ore types which were weathered appreciably. Unless alteration products were removed from the mineral particle surfaces, selectivity was rather poor. This condition had been noted previously in application of anionic flotation to these same ores.⁷ The principal effect of weathering on this cationic float was to inhibit flotation of stained particles of quartz and increase amine consumption. This substantially lowered the grade of concentrates obtained. It was found that conditioning at 65 to 70 pct solids for 5 to 10 min with 4 to 5 lb per ton of caustic followed by a decantation wash was effective in removing the alteration products on particle surfaces. When the spodumene-gangue separation

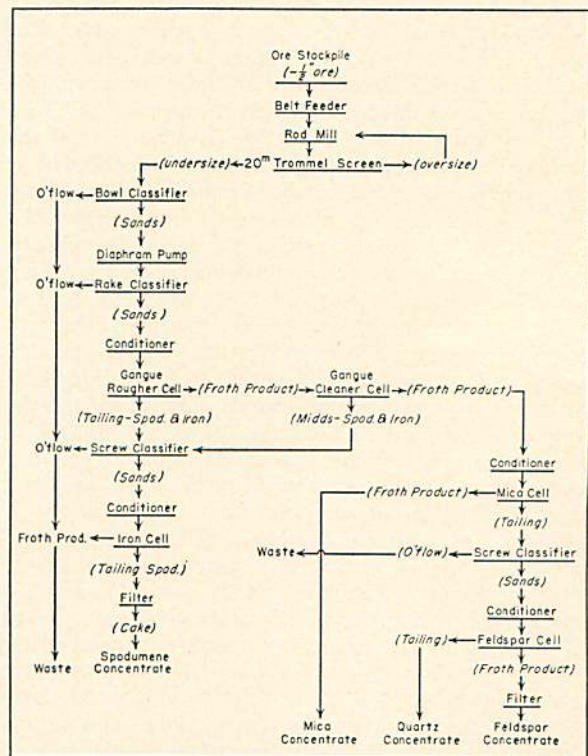


Fig. 1—Proposed spodumene flotation plant flowsheet.

Table III. Results of Batch Tests

Test 1 Dilute grind, 25 pct solids. No scrubbing.				
Item	Wt. Pct	Spodumene, Pct	Spodumene, Units	Distribution of Spodumene
Spodumene conc	20.0	71.0	14.3	65
Gangue froth	69.3	8.0	5.3	24
Slime loss	10.7			11
Total	100.0			100
Test 2 Dilute grind, 25 pct solids. Scrubbed 6 min at 75 pct solids with 5.0 lb per ton NaOH. Washed.				
Item	Wt. Pct	Spodumene, Pct	Spodumene, Units	Distribution of Spodumene
Spodumene conc	16.1	90.7	14.6	66
Gangue froth	72.9	5.4	3.9	18
Slime loss	11.0			16
Total	100.0			100
Test 3 Thick grind, 60 pct solids with 4.0 lb per ton NaOH in grind.				
Item	Wt. Pct	Spodumene, Pct	Spodumene, Units	Distribution of Spodumene
Spodumene conc	20.2	86.3	17.3	79
Gangue froth	66.3	5.0	3.3	15
Slime loss	13.5			6
Total	100.0			100

was performed on weathered ores which had been scrubbed in this manner, selectivity was usually as good as that obtained on fresh, unweathered ore samples.

An alternate method was also found to be effective. This alternate method was to change the grinding pulp density from low to high percent solids, using 4 to 5 lb per ton of caustic in the mill, thereby getting a scrubbing action in the mill. This procedure resulted in improved flotation selectivity, but changed the nature of the mill discharge from a rather coarse, uniformly-sized feed, to a finer feed with a much greater range of size.

Of these two methods of handling weathered ores, the one utilizing dilute grinding conditions and a separate scrubbing step has been shown to be the best procedure, although the alternative use of the mill as a scrubbing device may be used if it becomes expedient to do so.

The following three batch test results, see Table III, indicate the beneficial effect of scrubbing on a weathered ore. In each case shown, treatment of the samples was identical except for grinding and scrubbing conditions as indicated. Feed was Lab. No. 969.

Pilot Plant Tests

Since the method outlined in the preceding pages represented a novel approach to spodumene flotation, it was decided to make pilot plant tests so that the spodumene-gangue and spodumene-iron separations could be studied under continuous operating conditions. Part of the necessary equipment was furnished the laboratory by Foote Mineral Co.

The flowsheet followed was the same as that shown in Fig. 1, except that the gangue froth product was collected as a bulk froth product with no attempt made to separate the mica, feldspar, and quartz. Grinding conditions were set up to utilize the mill as a scrubbing device, i.e., high density grinding with caustic. Feed rate for most of the tests was 300 lb per hr of 1/2 in. ore, and a total of 15 tons of ore was used in these tests. Operating time on the test runs varied from 2 to 8 hr. Two ore samples were studied in this manner, Lab. No. 969 containing 22 pct spodumene, and Lab. No. 985 contain-

ing 12.5 pct spodumene. Seven complete tests were made on each ore. Variables studied in these 14 continuous pilot plant tests included amount of caustic used in the mill, pH studies in rougher and cleaner gangue floats, conditioning time and cell retention time in the gangue float section, comparison of pine oil, B-23, and cresylic acid as frothers, determination of optimum dextrine and amine amounts required, studies comparing sulphonated oils with soap as iron mineral collector, conditioning time and cell retention time in the iron float section. A short study was also made of the problem of concentration of the small amount of cassiterite which occurs in the spodumene ores. The cassiterite problem is not considered within the scope of this paper, but is mentioned to indicate that the presence of the mineral

Table IV. Results of Pilot Plant Tests

Test 2							
Operating Conditions							
Feed:—1/2 in. Lab. No. 969, 22 pct spodumene, 0.89 pct Fe ₂ O ₃ , fed at 300 lb per hr. Mill discharge —28 mesh.							
Equipment	Reagent	Quantity Reagents, Lb Per Ton	pH	Solids, Pct			
Rod mill	NaOH	4.34		65			
Rougher cell	NaOH	0.27	10.9	20			
	Dextrine	1.15					
	Amine	0.90					
	Pine oil	1.13					
Cleaner cell	Dextrine	1.10	10.0	15			
	Pine oil	0.69					
	Fe conditioner	Sodium resinate		3.20	5.0	65	
	HF	0.24					
Fe cell	Pine oil	0.76	5.4	20			
Metallurgical Results							
Product	Wt. Pct	Spodumene			Fe ₂ O ₃ , Pct	(1)	(2)
		Pct	Units	Dist.		Li ₂ O, Pct	Li ₂ O, Pct
Heads	100.0	22.0	22.0	100.0	0.89	1.90	1.30
Oversize	0.1	75.0	0.1	0.5		5.80	5.80
Bowl overflow	3.8	3.0	0.1	0.5	1.02	0.35	0.74
Rake overflow	3.2	3.0	0.1	0.5	0.68	0.35	0.87
Gangue froth	72.0	7.0	5.0	23.0	0.23	0.70	0.64
Iron froth	1.5	59.0	0.9	4.1	7.08	4.40	3.50
Spodumene conc	19.1	81.0	15.5	71.4	0.47	6.10	6.00
Total	99.7		21.7	100.0			
Test 14							
Operating Conditions							
Feed:—1/2 in. Lab. No. 985, 12.5 pct spodumene, 0.35 pct Fe ₂ O ₃ , fed at 325 lb per hr. Mill discharge —28 mesh.							
Equipment	Reagent	Quantity, Lb Per Ton	pH	Solids, Pct			
Rod mill	NaOH	4.80		65			
Rougher conditioner	NaOH	0.17	11.1	50			
	Dextrine	0.98					
	Amine	0.68					
	Pine oil	0.90					
	Rougher cell	NaOH		0.26	11.0	20	
Cleaner cell	Dextrine	0.98	10.5	15			
Iron conditioner	Pine oil	0.46					
	Sodium resinate	2.16					
	HF	0.70					
	Cresylic acid	0.52		65			
Metallurgical Results							
Product	Wt. Pct	Spodumene			Fe ₂ O ₃ , Pct	(1)	(2)
		Pct	Units	Dist.		Li ₂ O, Pct	Li ₂ O, Pct
Heads	100.0	12.7	12.7	100.0	0.35	1.15	1.15
Oversize	0.1	80.0	0.1	0.7		6.05	6.00
Bowl overflow	7.7	5.0	0.4	2.8	1.64	0.50	0.87
Rake overflow	2.3	6.0	0.1	0.7	0.96	0.60	0.81
Screw overflow	0.4	12.0			1.00	1.10	1.80
Gangue froth	77.0	4.7	3.6	26.0	0.19	0.50	0.42
Iron froth	0.6	53.3	0.3	2.2	11.00	4.30	3.96
Spodumene conc	11.8	80.0	9.3	67.5	0.65	6.05	6.44
Total	99.9		13.8	99.9			

(1) By using curve showing percent Li₂O vs percent sinks in acetylene tetrabromide.
 (2) By actual chemical analysis.
 Distribution based on percentage of sinks in acetylene tetrabromide.
 Chemical analyses by Foote Mineral Co.

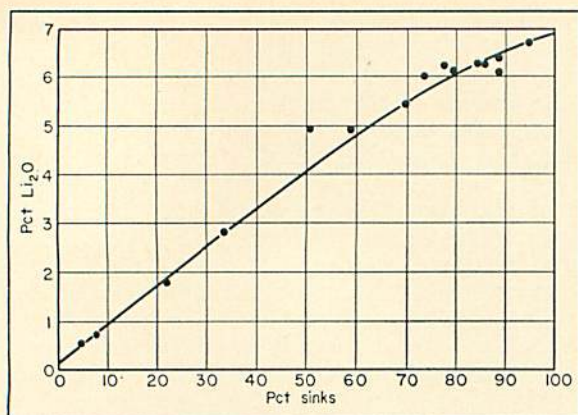


Fig. 2—Percentage of sinks in a given sample compared with percentage of Li_2O .

in interesting quantities was recognized and a method evolved to recover it.

Table IV shows the results obtained in pilot plant tests No. 2 and 14.

Heavy Liquid Control Method

The use of acetylene tetrabromide as a laboratory control aid is worthy of mention here in view of its extensive use in this investigation. The liquid has a specific gravity of 2.96. The only major constituent of the spodumene ores having a specific gravity higher than 2.96 was spodumene itself. It was found that -20 mesh ore samples, when placed in acetylene tetrabromide, resulted in a sinks product averaging 98 pct spodumene, the remaining 2 pct consisting of traces of cassiterite, horneblende, tourmaline, apatite, pyrite, and numerous others. Therefore, for all practical control purposes, the percentage of sinks in acetylene tetrabromide was the actual percentage of spodumene in the product.

A method was evolved in which the test samples were weighed, placed in acetylene tetrabromide contained in centrifuge tubes, and centrifuged for 10 min. Centrifuging brought about excellent separation of gangue minerals as floats and spodumene as sinks. Floats and sinks were then easily separated by a simple pouring technique, filtered, washed with benzene to remove excess tetrabromide, dried and weighed.

A graph, Fig. 2, was then drawn in which percentage of sinks in a given sample was plotted against percentage of Li_2O contained in the sample. Li_2O analyses for this graph were furnished by Foote Mineral Co. By obtaining the percentage of sinks in acetylene tetrabromide and reading the corresponding Li_2O value from the graph, it was possible to obtain a reasonably accurate Li_2O analysis.

This method was used as a control on laboratory batch tests and pilot plant tests. All products were quickly and easily evaluated from a lithia standpoint without the long and costly chemical analytical procedure for lithia determination. From 12 to 18 Li_2O determinations per 8-hr shift could be made by semi-skilled operators using this method. In addition to its use as laboratory control, the method could easily be adapted in commercial plant operating control.

Conclusions

The two flotation separations described in the foregoing pages produced spodumene concentrates from Kings Mountain ores on a continuous pilot plant scale. The spodumene concentrates contained an average of 6.0 pct Li_2O and 0.45 pct Fe_2O_3 with a

recovery of 70 pct available spodumene. If the iron removal step was not carried out the recovery of spodumene was approximately 75 pct. The process was successful on feed ground as coarse as 20 mesh. Weathered ores required light attrition scrubbing for satisfactory separation, but this was carried out in an acceptable manner by milling at high density with a high caustic addition.

The process is not recommended for use on ores containing less than 15 pct spodumene because recovery tends to drop off as spodumene content in the feed decreases. This is due to the fact that the gangue froth product inherently contains 3 to 5 pct spodumene, and this becomes an increasingly high percentage of the total distribution as the spodumene content of the ore decreases.

There were no particularly difficult operating problems. The two most important operating points, and the ones which might easily be overlooked, were proper pretreatment of weathered ores and strict maintenance of high pulp density in the conditioner before iron-mineral flotation.

The most difficult mineral to cope with was horneblende. Spodumene and horneblende are closely related mineralogically and exhibited similar reactions to the above described process. To remove horneblende satisfactorily, if it were encountered in appreciable amounts in the ore, it would probably be necessary to employ both flotation and magnetic separation.

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