Patented Oct. 2, 1945 2,385,819

UNITED STATES PATENT OFFICE

2,385,819

BENEFICIATION OF BERYLLIUM ORES

Frank D. Lamb, Hyattsville, and Lloyd Banning, Berwyn, Md., assignors to the Government of the United States, as represented by the Secretary of the Interior

No Drawing. Application September 13, 1943, Serial No. 502,081

6 Claims. (Cl. 209—166)

(Granted under the act of March 3, 1883, as amended April 30, 1928; 370 O. G. 757)

The invention described herein may be manufactured and used by or for the Government of the United States for governmental purposes without the payment to us of any royalty thereon in accordance with the provisions of the act of April 30, 1928 (Ch. 460, 45 Stat. L. 467).

This invention relates to the beneficiation of beryllium ores; and more particularly to the concentration of beryl from pegmatitic gangues.

It is an object of this invention to beneficiate beryllium ores. Another object is to concentrate beryl from ores containing pegmatitic gangues. Another object is to product beryl concentrates from low grade deposits to yield beryl concentrates of a marketable grade. Other objects will be apparent or will hereinafter appear.

These objects are accomplished in accordance with this invention by blunging a finely-ground, de-slimed beryl ore in admixture with aqueous alkali, thereafter adjusting the pH of the mixture to the approximately neutral range, then agitating and aerating the mixture in the presence of a frothing agent and collector comprising a fatty acid containing from 8 to 18 carbon atoms, and separating a froth containing the beryl concentrate.

It has been discovered that beryl can be concentrated from pegmatitic gangues of extremely variable composition by conditioning the finely-ground de-slimed ore with a strong alkali, thereafter the pH is adjusted to approximately the range pH 6 to pH 9, and by a flotation operation in the presence of a fatty acid collector and a frothing agent. It appears that the success of the operation is in some measure related to the alkali conditioning step followed by at least partial neutralization, since the high efficiency of the method appears to be influenced by this sequence of operations.

Suitable beryllium ores adapted for use in the present process include beryl when found in gangues of extremely variable composition, such as for example feldspar, clevelandite, quartz, mica, tourmaline, garnet, pyrrhotite, spodumene, zircon, pyrite, cassiterite, lithiophilite and purpurite. Other beryllium ores may also be adapted for concentration in the present invention, such ores as chrysoberyl, helvite and phenacite being exemplary, but the preferred beryllium ore for treatment is an ore containing beryl itself.

The ore is preferably stage-ground to minus 65 mesh (standard Tyler screen) and de-slimed by washing or other known means. Thereafter the ore is blunged for preferably 10 to 30 minutes in water containing per ton of ore from 1 lb. to 5 lbs. of caustic alkali, such as caustic soda, and the like. The blunging step appears to condition the ore for subsequent treatment. After blunging, the pH of the resulting pulp is adjusted to lie within the neutral to slightly alkaline range, such as pH 6 to pH 9, and preferably within the range pH 6.5 to pH 8. The adjustment of pH is made by neutralizing with a hydroxy aliphatic acid, such as glycolic acid, tartaric acid, citric acid and preferably lactic acid, or preferably by washing the ore with water until the pH lies within the desired range. The ore-pulp is then subjected to a froth flotation treatment in the presence of a frothing agent and a fatty acid, such as fatty acids of from 8 to 18 carbon atoms, for example, caprylic acid, lauric acid, oleic acid or stearic acid and preferably with a mixture of naturally occurring fatty acids obtained from coconut oil. Suitable frothing agents include lower alkyl glycolic esters, the mixture of branched chain higher synthetic alcohols produced in the methanol synthesis, and preferably the synthetic frothing agent known as "Emulsol X-1," believed to be the ammonium salt of lauryl diethylene glycol sulfate. The frothing agent can be employed in proportions of about 0.01 to 1.0 lb. per ton of ore and is preferably employed in proportions of about 0.05 to 0.5 pound per ton of ore.

The fatty acid reagent is employed in the proportions of 0.1 to 1 lb. per ton, and preferably from about 0.1 to 0.5 pound per ton of ore being treated. The flotation step can be carried out in any of the ordinary flotation apparatus, and the core is preferably conditioned by agitating with the flotation reagents from 5 to 25 minutes before the air feed is begun.

In a typical operation, a sample is stage-ground to pass a 20-mesh screen, being careful to avoid the production of fines. Thereafter the minus 20-mesh sample is further ground in a ball mill with an equal amount of water to minus 65 mesh size. The ground pulp is then de-slimed by admixing with 10 parts of water to each part of ore by weight, stirring and decanting the slimes. The resulting sands are agitated with an equal weight of water and from 1 to 5 lbs. of caustic soda per ton of ore. The agitation is continued for about 10 to 30 minutes, the charge then diluted with 10 parts of water per part of ore and the operation repeated until the pH lies within the preferred range of pH 6.5 to pH 8. Thereafter the pulp is diluted to 20% solids with water and flotation reagents added in the proportion of, per ton of ore, preferably 1 lb. of coconut fatty acid and 0.5 lb. of "Emulsol X-1." After a 5 minute condi-

tioning period the air is turned on in the flotation machine and the beryl concentrate recovered as froth over-flow. Preferably the flotation is carried out to produce a rougher concentrate, which is then passed on to a cleaner cell to produce a finished concentrate and middlings which are returned to the feed. Thus, one stage of flotation is carried on for recovery and the second stage for grade.

The following examples illustrate how the invention may be carried out but it is not limited thereto:

EXAMPLE 1

Beryllium ore from the Black Mountain deposit near Rumford, Maine, which assayed 1.7% BeO (or approximately 12% beryl) and which also contained feldspar (clevelandite), quartz and mica with minor amounts of tourmaline, garnet, pyrrhotite, spodumene, zircon, pyrite, cassiterite, lithiophilite and purpurite was treated in accordance with the foregoing outlined procedure.

The results are tabulated as follows:

TABLE 1.—RESULTS OF FLOTATION TEST ON BLACK MOUNTAIN ORE

Product	Weight, percent	Analysis percent beryl	Percent of total beryl
Cleaner concs	9.0	75.7	60.0
Cleaner tails	3.8	24.1	8.1
Rougher tails	58.9	1.9	10.0
Slimes	28.3	8.8	21.9
Composite heads	100.0	11.4	100.0

Pounds per ton of ore

Reagents	Blunge	Condition	Rougher	Cleaner
NaOH	4.0			
Coconut fatty acid		0.3		
Emulsol X-1		0.075		
Time, minutes	30	5	5	5

The results shown in Table 1 indicate a recovery of 60 percent of the total beryl with concentrates containing 75.7 percent beryl or 10.60 percent BeO. The greatest loss was in the slimes portion of the ore which contained 21.9 percent of the total beryl. Petrographic examination of the concentrates showed that the concentrates contain, beside beryl, mainly feldspar and mica, and minor amounts of tourmaline, spodumene, garnet, quartz, and pyrrhotite.

EXAMPLE 2

A beryl ore from the Weeks Quarry, East Wakefield, New Hampshire, which assayed 1.4% BeO (approximately 10% beryl) and in which the feldspar was in the orthoclase form, was similarly treated in accordance with the above outlined procedure. The results are tabulated as follows:

TABLE 2.—RESULTS OF FLOTATION TEST ON WEEKS QUARRY ORE

Product	Weight, percent	Analysis percent beryl	Percent of total beryl
Cleaner concs	8.3	87.0	74.7
Cleaner tails #1	2.0	1.0	0.2
Cleaner tails #2	1.3	25.0	3.4
Rougher tails	62.7	0.1	0.7
Slimes	25.7	7.9	21.0
Composite heads	100.0	9.7	100.0

Pounds per ton of ore

Reagents	Blunge	Condition	Rougher	Cleaner No. 1	Cleaner No. 2
NaOH	5.0				
Coconut fatty acid		0.8			
Emulsol X-1		0.4			
Time, minutes	30	5	5	5	5

Results obtained from the Weeks Quarry ore were somewhat better than those on the Black Mountain ore. Table 2 shows a recovery of 74.7 percent of the total beryl with concentrates assaying 87 percent beryl, or 12.2 percent BeO. Loss of beryl in the slimes amounted to 21.0 percent.

EXAMPLE 3

A beryl ore from the Gotta-Walden property, Portland, Connecticut, which assayed 0.55% BeO (3.9% beryl) and which contained considerable mica in the muscovite and biotite forms was treated in accordance with the above-outlined procedure. The results of the treatment are as follows:

TABLE 3.—RESULTS OF FLOTATION TEST ON GOTTA-WALDEN ORE

Product	Weight, percent	Analysis percent beryl	Percent of total beryl
Cleaner concs	4.1	94.0	71.4
Cleaner tails	0.7	5.0	0.6
Rougher tails	71.1	0.5	6.6
Slimes	24.1	4.8	21.4
Composite heads	100.0	5.4	100.0

Pounds per ton of ore

Reagents	Blunge	Condition	Rougher	Cleaner
NaOH	1.0			
Cocoanut fatty acid		0.8		
Emulsol X-1		0.4		
Time, minutes	30	5	5	5

Tests on the Gotta-Walden ore yielded higher grade concentrates than either the Black Mountain or Weeks Quarry ores. Results shown in Table 3 indicate a recovery of 71.4 percent of the beryl with concentrates assaying 94 percent beryl or 13.2 percent BeO.

EXAMPLE 4

The ore from the Weeks Quarry described in Example 2 was continuously treated by crushing in a jaw-crusher, then passing through rolls to pass a 6-mesh screen and thereafter separating the mica by screening through a vibrating screen. The ore then had the following characteristics:

TABLE 4.—RESULTS OF CRUSHING AND SCREENING WEEKS QUARRY ORE

Product	Size, mesh	Weight, pounds	Percent of total weight
Scrap mica	+6	45	6.3
Mill feed	−6	663	93.7
Composite		708	100.0

After screening to remove excess mica, the ore was ground in a ball-mill in closed circuit with an Akins type classifier. After grinding the ore was de-slimed in a Dorr Simplex classifier to produce a sized feed and slimes having the following characteristics:

TABLE 5.—SCREEN ANALYSIS OF PILOT PLANT SLIMES

Size, mesh	Percent of total weight	Cumulative percent
+200	1.2	1.2
−200+325	2.6	3.8
−325 sands	16.7	20.5
−325 slimes	79.5	100.0
Composite	100.0	

The de-slimed ore was then fed to a bank of six flotation cells made by the Denver Equipment Company and there blunged with caustic soda. The caustic soda was fed at the rate of 1 lb. per ton of ore, the pulp density in the machine averaged 1.20 and the pH was 11.5. After blunging, most of the caustic soda was washed out of the pulp by diluting with 10 parts of fresh water to one part of ore and pumping the pulp to a Dorr Duplex Rake classifier, where the over-flow went to waste and the raked product to a Denver conditioner. The coconut fatty acid and the "Emulsol X–1" was fed to the conditioner at the rate of 1 lb. of fatty acid and 0.5 lb. of "Emulsol X–1" per ton of ore. From the conditioner, the pulp flowed to the rougher cell of a bank of six flotation cells (Denver Equipment Co. type), froth from the rougher cell was returned to the cleaner cell and tailings from the rougher cell to three scavenger cells. Froth from the first cleaner cell was further cleaned in another cleaner cell and the tailings from both cleaner cells were returned as middlings to the rougher cell feed. The pH in the rougher cell was 7.2 and the pulp density therein was 1.13. A recovery of 69.5% of the beryl ore was made with concentrates containing 70.5% beryl. The results of the run are tabulated as follows:

TABLE 6.—RESULTS OF PILOT PLANT FLOTATION TEST

Product	Weight, percent	Analysis percent beryl	Percent of total beryl
Concentrates	4.9	70.5	69.5
Tailings	95.1	1.7	30.5
Composite heads	100.0	5.3	100.0

From the foregoing it is apparent that a desirable procedure has been provided for concentrating beryl from its ores, to yield a concentrate containing at least 9% by weight beryllium calculated as BeO.

Although the procedure is especially suitable for concentrating beryl from pegmatitic gangue materials, it is applicable to other beryllium ores as well.

Although in the examples, the slimes were discarded, they can likewise be treated to recover approximately 50% of the beryl therein by subjecting them to a similar pre-treatment with alkali and flotation.

Since many apparently differing embodiments of the invention will be apparent to one skilled in the art, various changes can be made in the invention without departing from the spirit and scope thereof.

What is claimed is:

1. A process for the beneficiation of beryl ores which comprises blunging a finely-ground deslimed beryl ore in an aqueous caustic alkaline pulp, thereafter adjusting the pH of the mixture to approximate neutrality, then agitating and aerating the mixture in the presence of a frothing agent and a collector comprising a fatty acid having from 8 to 18 carbon atoms, and separating a froth containing a beryl concentrate.

2. A process of concentrating beryl from its ores which comprises comminuting the ore to suitable particle size, pre-treating the comminuted ore by agitating it in a pulp containing from one to five pounds per ton of ore of caustic alkali, then washing the alkali away from the ore, then conditioning the thus alkali-treated and washed ore by agitating it in a pulp containing fatty acid and the ammonium salt of lauryl diethylene glycol sulfate, and subjecting the thus-conditioned pulp of beryl ore to froth-flotation to separate in the froth a concentrate of beryl.

3. A process of concentrating beryl from its ores which comprises comminuting said ore to at least minus 65-mesh particle size, pre-treating the comminuted ore by agitating it in a pulp containing from 1 to 5 lbs. of caustic soda per ton of ore, then washing the alkali away from the ore, then conditioning the thus-alkali-treated and washed ore by agitating for at least 10 minutes in a pulp containing flotation reagents consisting of coconut oil fatty acids and the ammonium salt of lauryl diethylene glycol sulfate and subjecting the thus-conditioned pulp of beryl ore to froth-flotation to separate in the froth a concentrate of beryl.

4. A process for the beneficiation of beryl ores which comprises blunging a finely-ground, deslimed beryl ore admixed with a pegmatitic gangue in aqueous caustic soda solution containing from 1 to 5 lbs. caustic soda per ton of ore, thereafter adjusting the pH of the mixture to the range pH6 to pH9, then agitating and aerating the mixture in the presence of a frothing agent in the amount of 0.05 to 0.5 lb. per ton of ore and about 0.1 to 1.0 lb. of coconut fatty acid per ton of ore and separating a froth containing a beryl concentrate.

5. A process for the beneficiation of beryl ore which comprises wet grinding pegmatitic beryl ore to minus 65-mesh size, de-sliming the ground ore, blunging the deslimed ore from 10 to 30 minutes with an equal weight of water containing from one to five pounds caustic soda per ton of ore, then diluting with excess water and decanting the solution until the pH of the pulp is between seven and eight, thereafter subjecting the pulp to agitation and aeration in the presence of, per ton of ore, about one pound of coconut fatty acids and about 0.5 pound of the ammonium salt of lauryl diethylene glycol sulfate, and separating a froth containing on the dry basis at least 9% by weight beryllium calculated as the oxide.

6. In a process for the production of a high-grade beryllium concentrate involving the flotation of a beryllium mineral, the steps which comprise pretreating a comminuted de-slimed beryllium ore with from one to five pounds per ton of ore of a strong alkali, adjusting the pulp pH to the neutral to slightly alkaline range, then agitating and aerating the pulp in the presence of coconut fatty acid and the ammonium salt of lauryl diethylene glycol sulfate, and separating a froth concentrate containing at least 9% by weight beryllium calculated as the oxide.

FRANK D. LAMB.
LLOYD BANNING.