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2,769,536

REAGENT CONDITIONING FOR ELECTROSTATIC SEPARATION OF BERYL

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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 me 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 separation of beryl from berylliferous materials, and, more particularly, to an improvement in the separation of beryl from pegmatite minerals by electrostatic methods.

The mineral beryl is difficult to separate by the usual separation processes from the minerals with which it occurs in nature, such as the quartz, feldspar, and muscovite occurring with beryl in pegmatite ores. Accordingly, separation of beryl by electrostatic separation methods has been suggested. However, electrostatic separation methods as heretofore employed have not resulted in an adequate recovery of beryl of a desired quality.

Accordingly, an object of this invention is to provide an improved method for separating beryl from associated minerals by electrostatic means.

Another object of this invention is to provide a method for increasing the selectivity of the electrostatic separation of beryl from other pegmatite minerals.

A further object of this invention is to provide a method for activating the mineral particles of berylliferous minerals whereby a more efficient separation of the beryl from its associated minerals may be accomplished.

These and other objects and advantages, hereinafter apparent as the ensuing description proceeds, are accomplished by this invention in a multistage conditioning operation which in general, comprises treating the beryl ore in an initial stage with an aqueous alkaline solution, treating the thus activated beryl ore in a further stage with an aqueous hydrofluoric acid solution, washing the conditioned ore with water containing emulsified fatty acids, drying the conditioned and washed ore at a low drying temperature, and subjecting the dried ore to electrostatic separation for the recovery of beryl.

Another embodiment of the invention comprises the use of multistage hydrofluoric acid conditioning of the beryl ore with omission of the alkaline treatment in the first stage of treatment. The hydrofluoric acid is an essential reagent in the conditioning process, but where used without alkaline pretreatment of the ore it must be employed in a plurality of stages as hereinafter described in order to provide sufficient activation of the ore for an effective electrostatic separation of the beryl. Both the multistage hydrofluoric acid treatment alone and in combination with a first stage alkali treatment yield an improvement in separation efficiency over the usual methods even with the emulsified fatty acid wash omitted. Use of the latter, however, further improves separation efficiency.

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When pegmatite ores containing beryl are conditioned wet in a single stage with several pounds of HF per ton of ore, washed with water, and then dried, some separation occurs on passing the ore through a static field type of electrostatic separator. The beryl is charged positive and the quartz and feldspar negative. Although separation does occur, the recoveries are low. The use of the multisage conditioning, combination of reagents, and conditions of the process of the present invention provides a greatly improved separation efficiency.

While the mechanism of the activation resulting in the improved separation is not definitely known, evidence indicates that the HF activation is a slime removal or surface de-adsorption mechanism. This is shown by the fact that a two stage HF conditioning using, for example, one pound of HF per ton of ore per stage is more efficient than a single stage conditioning using as much as four pounds of HF per ton of ore. In the two stage operation the ore is washed with water between stages.

Treatment of the ore in the initial stage with alkaline solution appears to render the HF activation more effective. For example, if a beryl ore is heated to 500° C., cooled, and then passed through the HF conditioning operation, no substantial separation results on passing the ore through an electrostatic separator. However, effective separation does occur if the first stage HF conditioning is replaced by an equivalent weight of a suitable alkali, such as NaOH or NH₄OH. The explanation of this phenomena is that surface alkali atoms, lost by volatilization at the 500° C. temperature, are replaced by absorption during the aqueous alkali conditioning.

It has also been found that beryl ores which have not been heated have a greater separation efficiency if the first stage of a multistage HF conditioning is replaced with an aqueous alkali conditioning step. This may be due to the fact that surface alkali atoms have been partially removed from the ore by weathering.

Ammonium hydroxide or any of the alkali oxides or hydroxides of the alkali group of the periodic table are satisfactory for the initial ore treatment. However, it is preferable to use NaOH since this is generally the least expensive.

The quantity of alkali and hydrofluoric acid required varies with respect to different ores. The HF required may range from 0.1 to about 10 pounds per ton of ore, and the optimum quantity for a particular ore may be determined by simple experiment. In general, one to two pounds of HF per ton of ore is sufficient. The amount of alkali to be used is approximately within the same range as the hydrofluoric acid.

The conditioning may be carried out with a pulp density within the range of from about 25 to about 75 percent solids. The conditioning agents conveniently may be added in solution in the water necessary to provide a pulp density within this range. Since part of the alkali and HF are removed from the liquid phase by adsorption on the ore particles, the actual content of the reagents in the liquid phase is variable during the treatment.

As aforesaid, the ore is washed between conditioning stages. Water may be used between stages for washing, but the separation efficiency is greatly improved when the final wash water, after the HF treatment, contains emulsified fatty acids. Any of the fatty acids which have filming characteristics, such as used in froth flotation are satisfactory. Examples of suitable fatty acids are coconut fatty acid and oleic acid. The fatty acid may be dispersed in the wash water with an emulsifying agent in a manner known to the art, or it may be added in the form of a soap which converts to an emulsified fatty acid on adjusting the pH to a value between about pH 6 and

pH 7. Examples of suitable soaps are those derived from animal and vegetable fats and petroleum and coal tar acids. These soaps frequently have a high content of stearic, palmitic, and naphthenic acids.

The preferred concentration of fatty acids in the wash water is approximately 0.006 percent of the weight of the water for an ore containing 0.5 percent or less of BeO. This amount is increased with increased BeO content. Thus, for an ore containing 1.5 percent BeO the concentration of fatty acid in the wash water may be increased to 0.01 percent. The optimum concentration may vary with the particular ore, but in general a concentration of less than 0.1 percent is satisfactory.

The separation efficiency is greatest when the pH of the final wash water containing the emulsified fatty acid is adjusted to a value near pH 7. The pH may be as low as about 6 or may be slightly above 7, but at higher values, as for example pH 9, the beryl fraction from the electrostatic separation is contaminated with the other pegmatite minerals. A mineral acid, such as HCl, may be used for adjusting the pH of the wash water to the proper value.

After the final wash, drying is necessary before electrostatic separation. The highest separation efficiency results when the drying is at a low temperature, as for example 100° C. If the drying is carried out at much higher temperatures the effect of the conditioning is completely lost. An electrostatic separator having a pure static field is satisfactory for the separation process.

As in all frictional electrical types of separations, a high moisture content of the surrounding atmosphere is detrimental. With the atmosphere at 30% relative humidity, and the feed to the separator at a temperature of approximately 50° C., satisfactory separation results. This moisture content of the air is equivalent to the absolute value of 40 grains of H₂O per pound of dry air. Moisture contents slightly higher or lower than this are also satisfactory.

The invention will be further illustrated by the following example of practice, but it is not intended to be limited thereto:

EXAMPLE I

The invention was applied as a two stage process to the conditioning of a beryl pegmatite from Maine containing 0.46 percent BeO. The beryl ore was crushed in a set of rolls and sized to obtain a minus 10 plus 35 Tyler mesh fraction. This fraction was then pulped and conditioned with aqueous NaOH solution amounting to 1 pound of NaOH per ton of feed and added with sufficient water to provide a pulp density in the range of from 25 to 75 percent solids. After agitation for a period of 5 to 30 minutes the mass was dewatered by draining, and washed with water and again drained. In a batch process washing must be repeated about four times. The same method of conditioning was then applied to the drained ore using aqueous HF instead of NaOH to the extent of 1 pound of HF per ton of feed. The ore was then drained and was washed with water containing 0.5 pound of soap per ton of feed and having a pH between 6 and 7, until the pH of the wash water drained from the ore showed a consistent pH within this range. The ore after the final draining was dried at a temperature below 100° C. and was then subjected to electrostatic separation. The feed was initially passed through a four roll separator with negative electrodes to yield a rougher reject and a rougher concentrate the latter of which was reprocessed over a six roll separator with positive electrodes. The six roll separator yielded a recleaned beryl concentrate, which was the fraction passing over the successive rolls, and a reclean reject. The muscovite is collected in the reclean reject along with the remainder of the quartz and feldspar not removed in the rougher reject. The results of the separation are shown in the table below:

Table I

TYPICAL ELECTROSTATIC SEPARATION OF BERYL PEGMATITE

Fraction		Weight, Percent	Assay, Percent BeO	Percent of Total BeO
No.	Class			
1	Rougher Reject	61.5	0.01	1.3
2	Reclean Reject	35.0	0.14	10.7
3	Beryl Concentrate	3.5	11.4	88.0
Composite		100	0.46	100

From Table I it may be observed that the BeO content was increased from 0.46 percent BeO in the initial ore to 11.4 percent BeO in the beryl concentrate, and that a recovery of 88.0 percent of the BeO content of the initial ore was made. This represents a practical recovery of BeO in a suitable concentration for further operations.

EXAMPLE 2

Beryl ore was crushed in a set of rolls to minus 10-mesh and was sized and deslimed by hydraulic classification so that two fractions, minus 10 plus 35-mesh and minus 35-mesh, were obtained. The sized ore was agitated from 5 to 30 minutes with NaOH and sufficient water to render the mass fluid and finally dewatered by draining. The drained ore was then washed with water and further drained. The washing and draining was accomplished by the batch method and accordingly were repeated at least four times. The same method of agitation and washing was now repeated using HF instead of NaOH, and wash water containing fatty acids instead of pure water. The ore after the final draining was then dried at a temperature close to 100° C. and immediately transferred to the electrostatic separator.

The process was carried out on a South Dakota ore containing 0.86 percent BeO. 1 lb. NaOH, 1 lb. HF and 0.5 lb. soap per ton of feed were used in the conditioning operation. The results of the separation are shown below:

Table II

Fraction		Weight, Percent	Assay, Percent BeO
No.	Class		
1	Rougher Reject	71.9	0.27
2	Reclean Reject	20.4	
3	Beryl Concentrate	7.7	10
Composite		100	0.86

EXAMPLE 3

When the reagents were increased to 2 lbs. NaOH, 2 lbs. HF and 1 lb. soap the ore and other conditions being the same as in Example 2 the results of Table III were obtained.

Table III

Fraction		Weight, Percent	Assay, Percent BeO
No.	Class		
1	Rougher Reject	70.5	0.066
2	Reclean Reject	22.4	0.54
3	Beryl Concentrate	7.1	10.7
Composite		100	0.86

It will be noted from Examples 2 and 3 that the increase in reagents improved the recovery and grade of beryl. The improvement with further increase of reagents is negligible. By visual examination the grade of the concentrate in Table II is shown to be less than that in Table III. A similar evaluation in further tests showed that the substitution of HF for NaOH in the first stage treatment

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yields results inferior to that of Table III, although adequate recovery for practical operation was obtained. Some ores apparently have a deficiency of alkali in the feldspar and associated minerals as a result of weathering. The NaOH has also the property of increasing the slime dispersion. Some slimes are more easily dispersed by alkalis than by acids and vice-versa. For best results the final conditioned and washed ore should have a minimum of slimes and a residual liquid having a pH close to 6 or 7, which is obtained by wash displacement and not by a neutralization of acid and base.

By means of the conditioning process disclosed, satisfactory separation of beryl by electrostatic operation may be accomplished where other methods of separation fail. A further advantage of the process is that it enables operation on crushed ore of large particle size which avoids slime losses encountered in other separation methods.

It will be appreciated from a reading of the foregoing specification that the invention described therein is susceptible of various changes and modifications without departing from the spirit and scope thereof.

What is claimed is:

1. A process for conditioning berylliferous ore for electrostatic separation of beryl from associated minerals comprising treating said ore in a first stage of a multistage conditioning operation with a dilute aqueous solution of an alkali, treating the ore in a further stage with a dilute aqueous solution of hydrofluoric acid and washing the ore between said stages.

2. A process for conditioning berylliferous ore for electrostatic separation of beryl from associated minerals comprising treating said ore with a dilute aqueous solution of sodium hydroxide, washing said treated ore, further treating said ore with a dilute aqueous solution of hydrofluoric acid, washing the thus-treated ore with water having a pH of about 7 and containing emulsified fatty acids

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until the pH of the wash water shows substantially no change and drying the washed ore at a low drying temperature.

3. A process for conditioning berylliferous ore for electrostatic separation of beryl from associated minerals comprising agitating the crushed ore with from 0.1 to 10 pounds of NaOH per ton of ore and sufficient water to render the mass fluid, dewatering and washing the treated ore, agitating the washed ore with from 0.1 to 10 pounds of HF per ton of ore and sufficient water to render the mass fluid, dewatering the treated ore washing the dewatered ore with water having a pH of about 7 and containing a small amount of emulsified fatty acid until the pH of the wash water shows substantially no change, and drying the washed ore at a temperature below about 100° C.

4. A process for the separation of beryl from associated minerals in a berylliferous ore comprising agitating the crushed ore with a dilute aqueous solution of an alkali, dewatering and washing the treated ore, further agitating the ore with a dilute aqueous solution of hydrofluoric acid, dewatering the treated ore, washing the ore with water having a pH of about 7 and containing a small amount of emulsified fatty acid, drying the washed ore at a temperature below about 100° C., and subjecting the dried ore to electrostatic separation.

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