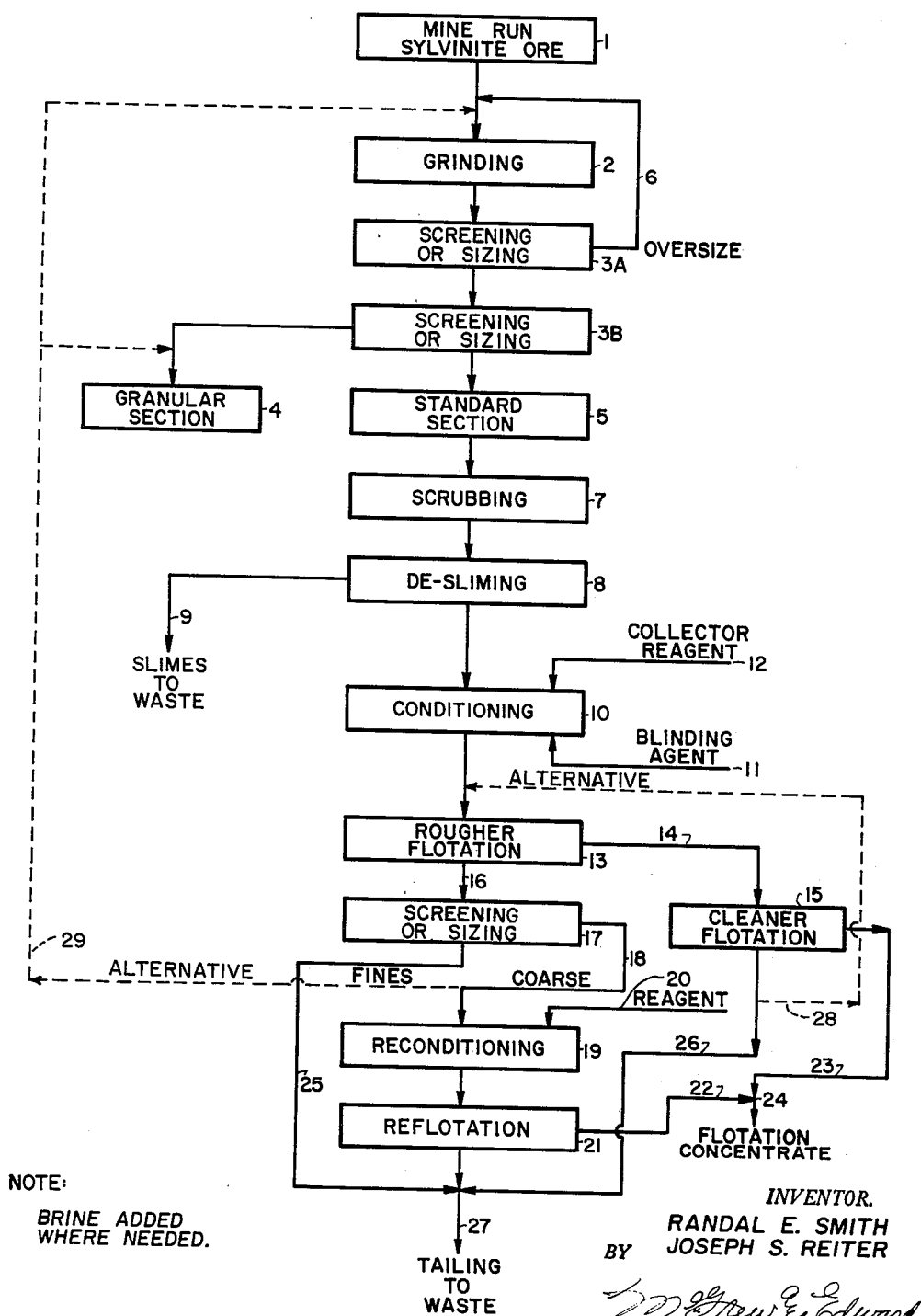


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REFLOTATION CONCENTRATION OF SYLVITE

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REFLOTATION CONCENTRATION OF SYLVITE

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This invention relates to froth flotation processes, particularly as applied to the treatment of sylvinite ores for the concentration of the sylvite content of such ores.

Flotation treatment of potash ores, particularly sylvinite ores, is highly developed, and commercial treatments have been devised to attain a high degree of concentration of the sylvite content of such ores, which concentrate is of good grade and comprises a marketable product as discharged from the treatment. The chief ores of sylvite are mixtures of halite and sylvite which may contain minor quantities of clay, polyhalite, anhydrite and other salts. Sylvite is the most valuable constituent. The present commercial practices for the treatment of sylvinite ores utilize a rougher-cleaner flotation section, and in the usual treatment the rougher concentrate constitutes the feed to the cleaner section.

It is well known that flotation tests conducted under ideal conditions such as in a laboratory or pilot plant will demonstrate that with optimum size control of the feed, reagent application methods, aeration, agitation rates, and treatment time, substantially complete flotation may be accomplished with very low collector reagent rates. It is also well known that it is very difficult to attain similar reagent rates under the usual refinery or concentrator conditions which are encountered in commercial operations.

In such commercial operations, if the rougher feed is conditioned with the amount of collector reagent which under ideal conditions will float substantially all of the sylvite content thereof, flotation will be incomplete particularly in the coarser sizes. To achieve satisfactory flotation of the coarse sizes under such conditions it is necessary to add substantially more collector reagent. With relation to the fine sizes, the additional reagent necessary to assure flotation of the coarse sizes is waste. This wasting of reagent may be reduced by one or more of the following practices, viz., careful size range control of the feed material, separate reagentizing according to size, variations in reagent makeup, or careful control of conditioning and flotation sections.

In the potash industry, it is noted that products in the coarser sizes are more desirable commercially. Tyler screen sizes are conventionally used in the industry to indicate product and feed sizes and are used throughout this specification. Granular potash in the range of about minus 6 plus 14 mesh carries a higher market price than the smaller sizes of equivalent grade. Consequently, it is advantageous to grind the ore feed so that it contains a high percentage of the coarser sizes. In many cases this type of grinding produces some true middling particles which are particles consisting partly of KCl (sylvite) and partly of NaCl (halite). Since the collector reagent is active only as to the sylvite portion of the particle, it will be seen that such particles are more difficult to float than particles of the same size consisting solely of sylvite.

The market advantage enjoyed by the coarser sizes has resulted in the preparation of ore feeds which contain particle sizes from about minus 6 mesh ranging downward. The larger sizes of about minus 6 plus 14 mesh which are difficult to float are more satisfactorily treated in a separate granular section, while the smaller, more easily floated sizes of about minus 14 mesh are treated in a separate section, usually called the standard section, having reference to the standard size of the material being

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treated therein. It will be noted that when, due to temporary failures in the ore preparation step, particles of larger size intended for the granular section are introduced into the standard feed, they will not float under the conditions established for the treatment of particle sizes intended to be floated in the normal operation of the standard section.

According to preferred practice of the present invention, the ore feed is prepared in the conventional manner by grinding in closed circuit to pass through a screen of about 6 mesh and then is passed through a sizing section where it is separated into two fractions, one designated "Granular" (about minus 6 plus 14 mesh) and the other designated "Standard" (about minus 14 mesh), after which the standard feed is scrubbed, deslimed, and conditioned with collector reagent in an amount necessary to float substantially all of the sylvite under optimum plant conditions. Alternatively, the ore feed may be scrubbed prior to sizing and deslimed prior to sizing. Whereas optimum flotation can be accomplished in the laboratory under ideal conditions with about .05 lb. amine per ton of ore feed, it will require about .1 lb. of amine reagent per ton of ore feed under optimum plant conditions.

After such conditioning the feed is subjected to rougher flotation treatment, and the rougher flotation concentrate product is sent to cleaner cells in conventional fashion. The finer sizes of the rougher underflow consist principally of halite with very little sylvite. The coarser sizes of the rougher underflow consist of coarse halite, coarse sylvite and coarse middlings. The middlings almost all occur in the coarser sizes. Such middling particles are more difficult to coat with collector reagent under refinery or concentrator operating conditions and will usually fail to float in the rougher flotation section unless substantially more collector reagent is added than would be necessary to float only the finer fractions.

We have found that the underflow from the rougher flotation section lends itself advantageously to mechanical separation to segregate the coarse sizes from the fine sizes. This may be conducted in any of the usual devices such as screening, wet classifying in conventional classifiers or in wet cone separators. We have found that the best results are obtained when the separation is at about plus or minus 20 mesh but the point of separation may be varied to suit the conditions encountered in actual practice. As used herein the minus 20 mesh fraction will be termed "fine sylvite" and the plus 20 mesh fraction will be termed "coarse sylvite." Separation should be made at a screen size where the finer fraction contains so little sylvite that it can be sent directly to tails. This will usually indicate a finer fraction containing not more than about 1% sylvite.

The classification applied to the rougher underflow, effectively separates the fine sized halite from the coarse sylvite, halite, and middlings so that in addition to performing the desired classification, the segregation of sizes amounts to at least a partial separation by chemical composition as well. The effect of this segregation is to reduce the volume of pulp being introduced into the later retreatment cycle so that only small capacity equipment is required for effective concentration at such stage. The reduced quantity of material being treated affords another benefit in that only a small quantity of amine reagent introduction is required in the reconditioning for the later retreatment cycle to insure high recovery of coarse sylvite and middlings.

After size separation of the rougher underflow into at least two fractions, the coarser fractions are reconditioned with collector reagent in amounts sufficient to float substantially all of the sylvite present and again treated in flotation cells. The unfloat fraction of this second flotation treatment will contain such a minor

amount of KCl that it is a satisfactory grade of salt for commercial purposes and may be taken as a market product when the operation is performed in an area where the requisite shipping costs of such product do not price it out of the market.

Accordingly, it is an object of this invention to provide a simple, economical and efficient process for the treatment of potash ores to recover high grade sylvite in size ranges providing a substantial quantity in segregated coarse sizes that are well suited for the market without blending or which may be blended with other high grade KCl in finer sizes to provide a product which is now recognized as an adequate coarse KCl product.

Another object of the invention is to provide a simple, efficient and economical flotation process for concentration of the sylvite content of the sylvinitic ores in which a high grade concentrate is obtained throughout the size range of the sylvite content with a minimum use of collector reagent.

A further object of the present invention is to recover the sylvite products which failed first flotation, whether due to improper feed preparation or improper flotation conditions.

Yet another object of this invention is to provide a novel method of flotation concentration of sylvite permitting preparation of a relatively coarse feed to the concentration circuit.

Other objects of the invention reside in the provision of novel steps and treatments for improving concentration in conjunction with reduced reagent consumption.

While the practice of the invention may utilize a variety of equipment including flotation cells, it is particularly advantageous to conduct the flotation at the refloatation stage in cells of the type shown and described in United States application Serial No. 838,544, and to operate said cells according to the process disclosure of United States Patent No. 2,931,502.

The practice of the invention will be best understood by reference to the accompanying flow sheet illustrating a typical arrangement of equipment and treatment steps utilized in the practice of the invention. As shown, ore, such as sylvinitic ore, from a suitable source of supply 1 is conducted through a grinding stage 2 and screening or sizing stage 3 (here shown as two stages 3A and 3B) to produce feed for separate flotation sections designated Granular Section 4 with feed about minus 6 plus 14 Tyler mesh and Standard Section 5 with feed about minus 14 Tyler mesh. If desired, screen oversize may be returned for regrinding as shown at 6. Referring to the feed to the Standard Section, the discharge from the screening stage 3 is subjected to scrubbing, if necessary, at 7, and desliming as indicated at 8 for removal of fine material. The materials removed as aforesaid usually have so little value that they are wasted as indicated at 9, but when they include sufficient KCl content to have commercial significance, such slimes may be passed to a subsequent treatment stage (not shown) for recovery of the sylvite content as brine.

Following removal of the slime content as aforesaid, the clean ore is subjected to a conditioning treatment 10 in which a clay blinding reagent and any of the well known amine collector reagents for sylvite may be introduced as shown at 11 and 12, respectively. Preferably, the quantity of such collector is limited to the amount required to float substantially all of the sylvite content under optimum plant conditions, usually about .1 lb. of amine collector per ton of ore feed. This amount will easily float substantially all of the fine fraction of the Standard feed (under about 20 mesh). The pulp after conditioning as aforesaid is introduced as feed to the rougher flotation section 13. The rougher concentrate is taken as feed 14 to the cleaner flotation section 15 in accordance with prevailing practice.

One of the innovations of this invention relates to treatment of the rougher underflow. This underflow 16 is

subjected to classification or screening, preferably screening on sieve bend units 17, and the oversize reject 18 of the screen is conducted to a reconditioning stage 19, and thence to the refloatation stage 21 as previously described. The fines discharge 25 of screening which is fine halite is passed to waste.

It will be observed that if the screening section 3 should malfunction and the feed to the standard section 5 should contain any granular material usually sized about plus 14 mesh, such granular material will probably not be floated at 13 but will report with the tailings 16 of rougher section 13 and will be separated at 17 for treatment at 19 and following stages. Enough additional amine collector is introduced at reconditioning stage 19 as shown at 20 to insure flotation of substantially all sylvite fractions, but inasmuch as such material has received a substantial coating or filming in the initial conditioning stage 10, only a slight addition to particle surfaces is necessary to render the coarse particles readily floatable. Consequently, the total reagent introduction at conditioning steps 10 and 19 is substantially less than would be required if an attempt were made to float all sylvite sizes in the rougher flotation stage.

The flotation treatment at the coarse flotation stage 21 may be performed in any suitable apparatus, such as standard type mechanical flotation cells, but as noted previously, is preferably performed in a jet cell of the type shown in Patent No. 2,931,502. Most of the stages described above are carried out with the ore pulped in brine saturated as to sylvite and halite. Brine will therefore be introduced or removed as required at various stages of the process. These are standard procedures and are not indicated on the flowsheet but a note about brine addition is shown. The refloatation underflow comprising halite is passed to waste as shown at 27.

The concentrate 22 of refloatation stage 21 may be leached for upgrading (not shown) before being discharged from the process as product, preferably going through the dewatering stage by which the brine is separated and held in the plant circuit. The final stage of the dewatering procedure will be a drying stage (not shown) so that the final product passing from the plant is sufficiently dry to permit storage or transportation as required.

The concentrate 23 of the cleaner stage 15 provides another marketable sylvite product which is also of high grade, but in the standard flotation size range. In usual practice, this will be discharged from the plant through a dewatering section of conventional type (not shown) and may be handled as a separate product for sales purposes. However, all or a part of such concentrate may be conducted for blending with the coarse KCl product as shown at 24 and in the present plant practice, it is customary to have a number of such blends involving different size ranges, all of which are commercial products for particular purposes. The cleaner underflow may be conducted to waste as shown at 26, recycled to flotation as shown at 28, or otherwise retreated.

In the preceding description, reference has been made to the introduction of additional collector reagent at 20 in the conditioning ahead of the coarse refloatation stage 21. Usually the collector reagent will be the same composition as introduced into the conditioning stage 10, but for certain types of ore, it may be preferable to include a different amine or an amine-oil mixture as the collector reagent introduced at reconditioning stage 19.

In order to illustrate the efficiency of the flotation of sylvinitic according to this invention, a typical test operation is reported. Sylvinitic ore from the Carlsbad basin was used. Screen analysis of the ore and of products is shown in the following chart or tabulation.

Ore weighing 381 grams was pulped with brine and subjected to a desliming treatment for removal of clay and other slime constituents. In this operation, 25 grams of slimes were removed. The cleaned ore was then con-

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ditioned with 0.5 lb. of starch and 0.1 of octadecyl amine per ton of ore and a little frother oil. On rougher flotation, 120 grams of concentrate were produced as a floated product and 236 grams were produced as unfloats residue (rougher underflow). The 236 grams of unfloats residue (rougher underflow) were screened roughly on a 20 mesh screen producing 135 grams of minus 20 mesh material and 101 grams of plus 20 mesh material.

The 101 grams of plus 20 mesh material were then reconditioned with .15 lb. of amine and 0.8 lb. of a heavy fractionator-bottom oil per ton of this coarse flotation feed. On refloatation of the plus 20 mesh material, 22.3 grams of coarse concentrate were produced as the floated product and 78.8 grams of coarse tailings were produced as the unfloats residue.

Screen analyses of value in interpretation of the results of such test are presented in the following chart. Tyler mesh are used and the proportion on each mesh is given as a percentage.

Mesb.....	10	14	20	28	35	48	65	100	150	200	-200
Feed.....	9.3	12.8	18.9	14.7	10.5	10.6	7.7	6.2	2.7	4.5	2.1
1st Concentrate.....	0.6	5.9	17.4	17.8	13.9	14.8	9.8	9.9	4.2	3.4	2.3
Rougher underflow (R.U.).....	12.4	13.9	18.1	14.1	10.1	10.1	6.5	6.7	3.5	2.9	1.7
Minus 20 mesh (R.U.).....			0.2	19.6	18.5	17.0	14.6	12.3	8.5	5.1	4.2
Plus 20 mesh (R.U.).....	26.3	29.5	38.1	6.1							

In carrying out the operation the following weights and grades expressed as K_2O content were found:

	Weight in Grams	Percent K_2O	Weight K_2O in Grams	Percent KCl Distribution
Feed Ore.....	381	23.90	91.07	100
Slimes.....	25	20.64	5.16	5.6
1st Concentrate.....	120	59.62	71.54	78.6
Rougher Underflow (R.U.).....	236	6.09	14.37	15.8
Minus 20 Mesh (R.U.).....	135	1.01	1.36	1.5
Plus 20 Mesh (R.U.).....	101	12.88	13.01	14.3
Coarse Concentrate From R.U.	22.3	54.56	12.17	13.4
Coarse Tailings From R.U.	78.8	1.06	0.84	.9

On scrubbing the ore for release of slime, some breakage occurred, but it will be noted that the first concentrate was considerably finer in size than the feed and that the rougher underflow was coarser. On screening the rougher underflow, the fine fraction (minus 20 mesh) was of low grade while the coarse fraction (plus 20 mesh) carried 12.88% K_2O . This is a fairly good grade of feed, and on refloatation, a coarse tailings carrying 1.06% K_2O was produced.

Therefore on a coarse feed and with a minimum consumption of reagent which calculates to 0.14 lb. of amine per ton of ore the recovery was 78.6 and 13.4 or 92% of the potash in the ore. Tailings account for 1.5 and 0.9 or 2.4% of the potash. The chief loss is with the slimes which can be recovered by conventional solution methods.

In operation, the first concentrate of grade 59.62 would be refloats in a cleaning stage to bring it up above 60.00% K_2O the present specification on marketable fertilizer potash. The grade of the coarse material which contains appreciable middling material was 54.56% K_2O and this would be leached to bring it up to grade also. However, by this separation, the leaching operation is carried out on only a small proportion of the total concentrates.

Present commercial practice has established that in order to float material of the large or coarse sizes floated in the test reported above at a comparable low tailings rate, the reagent requirement would be from 0.2 lbs. amine per ton of ore treated to several times that amount, and would apply to the total tonnage of feed to the floata-

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tion section. Thus, in a plant treating several thousand tons of ore per day, a significant reduction in reagent cost can be effected while maintaining a high efficiency in the concentration.

As an alternative procedure, sizing section 3B may be omitted and granular section 4 bypassed and all feed sent to standard section 5. In this event, the rougher underflow will contain substantial sylvite values in the coarser sizes. The coarse fraction of the rougher underflow 18 may be treated as indicated at 19 and 21, or may be used as feed to granular section 4, or it may be reground and returned to rougher flotation 13 with or without additional conditioning as indicated by the alternative circuiting 29.

As another alternative, sufficient collector reagent may be added at conditioning stage 10 to float substantially all of the sylvite (whether or not a size separation is performed at 3B). Flotation is then conducted by carefully controlling the aeration or agitation rate, or both, in

rougher flotation 13 so that substantially all of the sylvite in the size range of about minus 20 mesh is recovered as flotation concentrate overflow product. Coarse fraction 18 may then be treated as indicated at 19 and 21, or subjected to more thorough aeration and agitation in additional or continuing flotation cells.

As still another alternative, if sufficient collector reagent has been added at the conditioning step 10 prior to rougher flotation, reconditioning at step 19 may be with a water-immiscible oil followed by an additional or further flotation treatment. Other changes and modifications may be availed of in the selection and arrangement of equipment utilized in the practice of this invention, and in the procedures at the several treatment stages, within the spirit and scope of the invention as defined in the hereunto appended claims.

We claim:

1. In a continuous froth flotation process for the concentration of the sylvite content of sylvinitic ore, in which a deslimed pulp of such ore is subjected to a rougher flotation step utilizing a collector reagent for sylvite, the improvement which comprises separating substantially all the fine sylvite of about minus 20 mesh sizes of the deslimed pulp as a froth concentrate in the rougher flotation step, subjecting the underflow solids of said flotation step to a size separation step for segregation of a fraction of about plus 20 mesh sizes, reconditioning said plus 20 mesh fraction so segregated with a sylvite collector, subjecting the pulp so conditioned to another flotation step, and recovering most of the coarse sylvite content of about plus 20 mesh sizes in the rougher flotation underflow as a concentrate of the latter flotation step.

2. In a continuous froth flotation process for the concentration of the sylvite content of a sylvinitic ore, in which a deslimed pulp of such ore is subjected to a rougher flotation stage, the improvement which comprises conditioning said pulp with an amine collector reagent for sylvite in an amount required to float substantially all of the sylvite content except coarse sizes of about plus 20 mesh at said rougher flotation stage, subjecting the underflow solids of said rougher flotation stage containing unfloats sylvite to a size separation step for segregation of a fraction of about plus 20 mesh sizes containing substantially all of the unfloats sylvite of the feed to the rougher flotation step, reconditioning the plus 20 mesh fraction so segregated with an amine collector

in an amount sufficient to float substantially all of the remaining sylvite content, introducing the pulp so reconditioned into a second flotation stage, and recovering most of the coarse sylvite content of about plus 20 mesh sizes in the rougher flotation underflow as a concentrate of the latter flotation stage.

3. In a continuous froth flotation process for the concentration of the sylvite content of sylvinite ore, in which a deslimed pulp of such ore is subjected to a rougher flotation step utilizing a collector reagent for sylvite, the improvement which comprises separating substantially all the fine sylvite of about minus 20 mesh sizes of the deslimed pulp as a froth concentrate in the rougher flotation step, subjecting the underflow solids of said flotation step to a size separation step for segregation of a fraction of about plus 20 mesh sizes, reconditioning the plus 20 mesh fraction so segregated with a sylvite collector, subjecting the pulp so conditioned to a second flotation step, recovering most of the coarse sylvite content of about plus 20 mesh sizes in the rougher flotation underflow as a concentrate of said second flotation step as a final flotation product, subjecting the rougher concentrate to a cleaner flotation step, recovering a sylvite concentrate of said cleaner flotation step, and combining said fine concentrate with said coarse sylvite concentrate as a final sylvite product.

4. In a continuous froth flotation process for the concentration of the sylvite content of sylvinite ore, in which a deslimed pulp of such ore is subjected to a rougher flotation step, the improvement which comprises conditioning said pulp for said flotation step with a collector reagent for sylvite to float substantially all said sylvite content except coarse sizes of about plus 20 mesh, separating substantially all the fine sylvite of about minus 20 mesh sizes of the conditioned pulp as a froth concentrate in the rougher flotation step, subjecting the underflow solids of said rougher flotation step to a size separation step for segregation, removal and discard of a fine fraction containing a minor portion of the sylvite in said underflow solids from a coarse fraction of about plus 20 mesh sizes, reconditioning the separated coarse fraction with a collector in an amount sufficient to float substantially all of the remaining sylvite content, subjecting the pulp so reconditioned to a second flotation step, and recovering most of the coarse sylvite content of about plus 20 mesh sizes in the rougher flotation underflow as a concentrate of said second flotation step.

5. A continuous froth flotation process for the con-

centration of the sylvite content of sylvinite ore, which comprises subjecting a deslimed pulp of sylvinite ore in a mixed size range of about minus 14 mesh to a rougher flotation step utilizing a collector reagent for sylvite for separating substantially all the sylvite content except coarser sizes of about plus 20 mesh as a concentrate, subjecting the underflow solids of said flotation step containing unfloated coarse sylvite to a size separation step for segregation of a fraction of about plus 20 mesh sizes containing substantially all of the unfloated sylvite of the feed to the rougher flotation step, reconditioning said plus 20 mesh fraction with a sylvite collector, and subjecting the reconditioned pulp to another flotation step for recovering most of the unfloated sylvite in the rougher flotation underflow as a concentrate of the latter flotation step.

6. A continuous froth flotation process for the concentration of the sylvite content of sylvinite ore, which comprises subjecting a deslimed pulp of sylvinite ore in a mixed size range of about minus 14 mesh to a rougher flotation step utilizing a collector reagent for sylvite for separating substantially all the sylvite content except coarser sizes of about 20 mesh as a concentrate, subjecting the underflow solids of said flotation step containing unfloated coarse sylvite to a size separation step for segregation of a fraction of about plus 20 mesh sizes containing substantially all of the unfloated sylvite of the feed to the rougher flotation step, reconditioning said plus 20 mesh fraction with a sylvite collector, subjecting the reconditioned pulp to another flotation step for recovering most of the unfloated sylvite in the rougher flotation underflow as a concentrate of the latter flotation step, and combining the concentrate of both flotation steps as a final sylvite product in the size range of the feed to the rougher flotation step.

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