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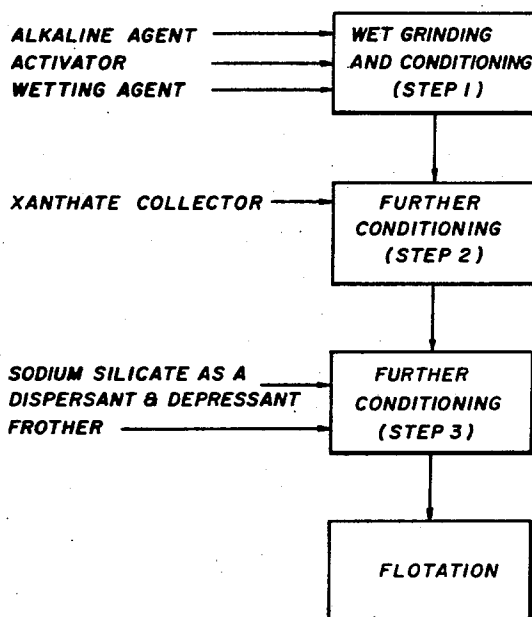
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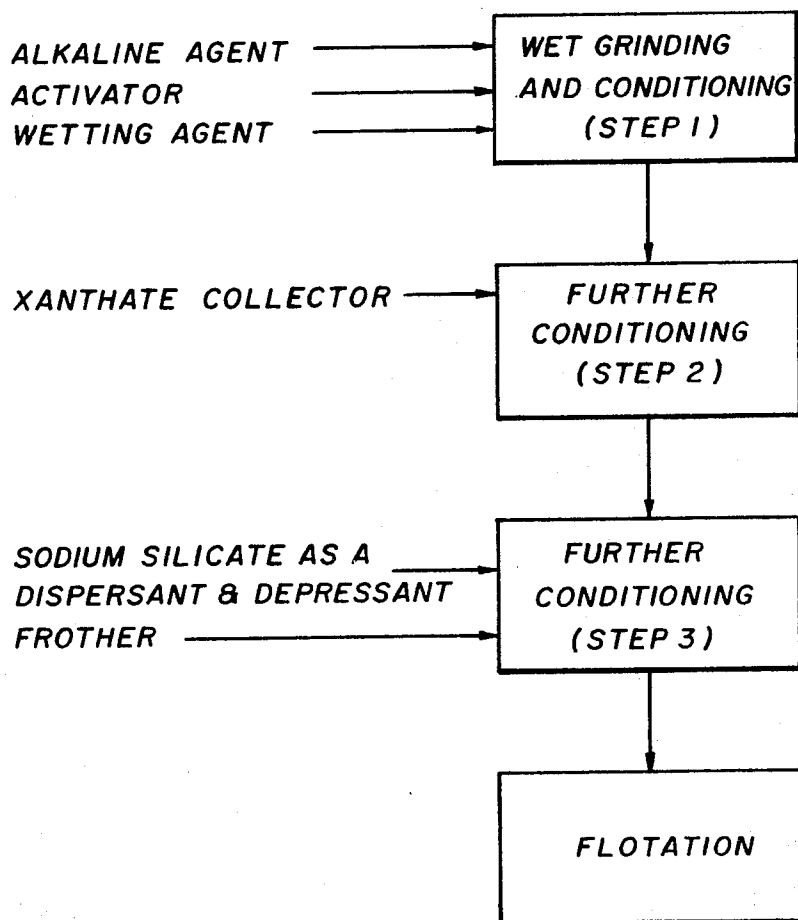
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ABSTRACT: Flotation of sulphide minerals from their ores is achieved by a process in which two or more conditioning steps are carried out prior to a froth flotation step. The first step comprises conditioning a pulp of the ore in the presence of at least one alkaline agent selected from a group of agents which is defined in the specification which follows. The pulp is then further conditioned in the presence of a member of the xanthate family of flotation reagents and sufficient sodium silicate to effectively disperse the host rock minerals. The resulting pulp is then subjected to froth flotation. Preferably there are three separate conditioning steps, namely conditioning carried out in the presence of (1) one of the hereinafter defined alkaline agents, (2) a member of the xanthate family of flotation reagents, and (3) sodium silicate. The invention is particularly valuable for the recovery of metallic sulphides from ores containing talcy minerals or natural slimes and the invention has been applied with signal success to the flotation of copper-nickel sulphide ores containing over 40 percent by weight of talcose host rock minerals.





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FLOTATION OF SULPHIDE ORES

BACKGROUND OF THE INVENTION

This invention relates to the flotation of sulphide minerals from their ores and is especially valuable in the case of sulphide ores containing appreciable quantities of talcy-type host rock minerals. In prior flotation procedures with appreciable amounts of such host rock minerals, large quantities of the host rock are floated with the sulphides, thereby making the operation uneconomical or resulting in low grade concentrates which are difficult or impossible to upgrade, and, in many cases, poor recoveries of the sulphide values.

In addition to the above-described ores, generally referred to as talcose ores, this invention is especially useful for the recovery of sulphides from a broad field of host rock materials such as those contained in mud seams, graphite, and ores with natural slimes that interfere generally in the same manner in the flotation of sulphide ores containing talcose-type minerals.

In the past, the method of treating such ores has been either to deslime the pulp before flotation resulting in major sulphide value losses, or a prefloat made ahead of the sulphide float also resulting in major sulphide losses, or where the ore is of sufficiently high grade the deleterious material is floated with the sulphides resulting in a low grade concentrate being shipped to the smelter, or alternately, cleaned many times, resulting in a medium grade concentrate with comparatively low value recoveries.

BRIEF SUMMARY OF THE INVENTION

I have invented a process wherein the sulphides can be directly floated at a moderate cost from such ores, with the production of high grade concentrates and high sulphide value recoveries.

With my process there is no need to deslime prior to the sulphide flotation, or make an initial prefloat for discarding to tailings or further treatment.

As will be seen from the following, my process may involve unusual combinations of reagents together with preconditioning steps wherein the reagents added in each step and their concentrations are major factors in this surprisingly effective flotation procedure. It will also be seen in the following that selection of alkaline agents is most important and optimum results are only obtainable with correct combinations of agents.

My invention may be generally defined as a process for the flotation recovery of at least one sulphide mineral from an ore of the same by a process in which at least two stages of conditioning precede flotation, said process comprising conditioning a pulp of the ore in the presence of an alkaline agent selected from the group consisting of alkali metal carbonates, ammonium hydroxide, mixtures of these compounds, and mixtures comprising at least one of the compounds and at least one hydroxide selected from the group consisting of sodium, potassium and calcium hydroxides; and, in the presence of a member of the xanthate family of flotation reagents and a sufficient amount of sodium silicate to maintain the host rock minerals effectively dispersed, further conditioning the said pulp and subjecting same to froth flotation.

The presently preferred process according to the invention includes three conditioning steps. In the first conditioning step the ore, together with activating agents or wetting agents which are to be used, is conditioned in the presence of an alkaline agent which may be either an alkali metal carbonate or ammonium hydroxide, or mixtures of these compounds, or mixtures of one or both of these compounds with a hydroxide selected from the group consisting of sodium, potassium and calcium hydroxide. This conditioning step is carried out for a period of time sufficient to activate the mineral and with sufficient alkaline agent to bring the pH of the resulting pulp to a point within the optimum range for flotation of the desired mineral. The pulp resulting from this first step is now subjected to a second conditioning step in the presence of a

selected member of the family of xanthate flotation reagents for a time predetermined by experiment to produce maximum activation. The third conditioning step comprises conditioning the pulp resulting from the second step in the presence of sodium silicate once again in an amount and for a time predetermined by experiment to be sufficient to effectively disperse the host rock minerals. The pulp resulting from the third conditioning step is then subjected to froth flotation.

Further, as will be seen from what follows, the time of flotation required after the stages of preconditioning is comparatively short as compared to conventional flotation circuits, even as compared with those which are free of such deleterious material. This not only means an appreciable saving in capital cost but also a possible reduction in operating costs. Following the addition of the frother I found that the sulphides float in the rougher circuit extremely rapidly using a rougher flotation time of 7 to 12 minutes. The normal rougher flotation time in the flotation of the nickel mineral pentlandite in current conventional circuits is 45 minutes.

The data from this invention also indicates that it may be applied for additional host rock rejection to current base metal sulphide flotation plants to reduce the insol in the final concentrates now being produced from ores containing the normal host rock materials and that have heretofore not been considered troublesome. By reducing the insol in the concentrates both freight and smelter costs will be reduced.

In the detailed description which follows, I have shown that excellent results may be obtained on a copper-nickel sulphide ore using as one combination, sodium carbonate and ammonium hydroxide as the alkaline controlling reagents in the process. In the application of this process, it may well be found that on other types of ores containing different host rock constituents or other sulphides such as lead and zinc, other alkaline combinations may either be more economical or show improved results over the sodium carbonate-ammonium hydroxide circuit found best for this copper nickel ore. With this copper nickel ore I have found it advantageous to use copper sulphate as an activator in the initial conditioning step, whilst with a sulphide such as galena no such activating agent is required.

When the conventional xanthate flotation method for flotation of this ore is used, the inventor was informed that the maximum grade of concentrate producible was in the range of 2—2½ percent nickel with recovers of approximately 65—70 percent. With my process a concentrate grade in excess of 5 percent nickel was produced with final recoveries indicated in excess of 80 percent.

In my process I not only found that the preconditioning stages were important, but also the method in which I carried out my cleaning. Again, a short conditioning stage with the addition of reagents before each cleaner gave optimum results.

BRIEF DESCRIPTION OF DRAWING

A drawing is attached which is a flow sheet of the preferred embodiment of the invention.

DETAILED DESCRIPTION

Referring to the drawing, in this preferred procedure which is applied to the flotation of copper and nickel sulphides from its ore, the wet grinding and first-conditioning step are carried out simultaneously, the ore being fed to a wet-grinding system together with a wetting agent, at least one alkaline reagent, and an activator for the nickel sulphide minerals, namely copper sulphate. When the ore has been ground to the desired fineness and the sulphides activated, the ore is taken from the grinding circuit and subjected to a second conditioning step in the presence of a selected member of the xanthate family of flotation reagents. This treatment is carried out for a period which must be predetermined by experimentation in a manner which will be described in detail later. The thus conditioned ore is subjected to a further (third) conditioning step in the presence of an amount of sodium silicate sufficient to effec-

tively disperse and depress the host rock minerals. A conventional frother is added toward the end of the third-conditioning step and the resulting pulp subjected to froth flotation.

A detailed description of the preferred procedure of the invention is set out in example I which follows. The ore treated in example I was a copper-nickel sulphide ore containing an estimated average 40 percent of talcose-type minerals. The sample contained 0.52 percent nickel in the form of pentlandite and 0.025 percent copper in the form of chalcopyrite. The ore was ground in a laboratory rod mill at 60 percent solids to a fineness of about 95 percent minus 200 mesh in the

bonate in combination with lime alone and sodium carbonate in combination with sodium hydroxide. Because of the similar chemical properties of sodium hydroxide and potassium hydroxide, it is apparent that potassium hydroxide could be substituted for sodium hydroxide in the combinations above where sodium hydroxide gave beneficial results. I have set out below in tabular form the results of a series of tests using various alkaline reagents, the conditions in each case being exactly the same as described in example I except for the indicated changes in the alkaline reagent (s) employed.

The test numbers are shown in the first column of this table

TABLE I
Table Showing Effect of Use of Different Alkaline Reagents

Test No.	pH Step 3	Alkalis used, lbs./ton				Rougher tailings		
		Na ₂ CO ₃	NH ₄ OH	Ca(OH) ₂	NaOH	Percent wt.	Percent Ni	Percent Cu
204.....	9.8	7.5	0.62	0.0	0.0	75.7	0.097	0.018
213.....	9.5	0.0	0.62	5.0	0.0	73.1	0.107	0.019
214.....	9.8	0.0	0.62	7.5	0.0	68.7	0.116	0.019
215.....	9.7	0.0	0.0	6.25	0.62	72.9	0.123	0.019
216.....	9.85	3.75	0.62	3.75	0.0	69.6	0.096	0.019
218.....	9.75	7.5	0.0	0.0	0.62	74.9	0.107	0.019
219.....	9.7	8.1	0.0	0.0	0.0	74.5	0.101	0.019
223.....	9.85	0.0	0.0	0.0	3.0	75.0	0.113	0.019
224.....	9.4	0.0	4.25	0.0	0.0	73.8	0.104	0.018
228.....	9.35	0.0	0.0	6.25	0.0	67.2	0.119	0.019

presence of the equivalents of 7.5 pounds per ton of ore of sodium carbonate, 0.62 pounds per ton of ore of ammonium hydroxide, 0.45 pounds per ton of ore of activator (copper sulphate) and 0.075 pounds per ton of ore of a wetting agent which was a trimethyl nonyl ether of polyethylene glycol. The grinding time was 40 minutes during which period the first conditioning step was completed. The resulting pulp was then subjected to a second conditioning step for a period of 20 minutes with the addition of 0.125 pounds per ton of potassium amyl xanthate. The pulp resulting from the second-conditioning step was subjected to a third-conditioning step for a period of 10 minutes in the presence of 35 pounds sodium silicate per ton of ore. At the end of the second step the sulphides showed heavy flocculation at the surface of the pulp. Following the addition of sodium silicate in the third step, the host rock was heavily dispersed while the sulphide flocs only partially broke up. At the end of approximately 1 minute following the addition of sodium silicate the sulphide flocs strongly reformed while the host rock minerals remained heavily dispersed. The pH of the pulp at the end of the third-conditioning step was 9.8. During the last 2 minutes of this conditioning stage, frother was added which was two drops of pine oil. The resulting pulp was then subjected to froth flotation for a period of 12 minutes during which 0.05 pounds per ton of xanthate was added and two additional drops of pine oil. The pulp temperature was 36° C.

The rougher tailings amounted to 75.6 percent by weight and were remarkably low in nickel content. The nickel analysis was 0.097 percent while the copper analysis was 0.018 percent. So far as I am aware, it has never in the past been possible to obtain results of this order in the case of an ore having a talcose content of this magnitude.

While in the preferred process described in detail above all of the first conditioning step is completed during wet grinding it should be appreciated that the first step might be carried out partially during wet grinding, with the remainder of this first conditioning step being conducted after the grinding has been completed. Furthermore, in certain cases it may be desirable to carry out the entire first-conditioning step after either wet or dry grinding.

As indicated above, other alkaline reagents may be employed but experiments I made with the conventional alkaline reagent used in the flotation of copper and nickel sulphides namely lime, gave poor recoveries and had a tendency to float an appreciable percentage of the host rock. The use of sodium hydroxide by itself also gave unsatisfactory results. I was, however, able to obtain satisfactory results with, in addition to the combination mentioned in example I, sodium carbonate by itself, ammonium hydroxide by itself, sodium carbonate and ammonium hydroxide in combination with lime, sodium car-

and test No. 204 is the test which gave the results fully set out above in example I.

It is immediately apparent from a study of table I that the results obtained with calcium hydroxide alone, sodium hydroxide alone or a combination of these two components gave inferior metallurgical results. Throughout this table the illustrations shown represent the best metallurgy that was attained in optimizing such reagent or reagent balances.

Reference will now be made to the manner in which one may predetermine the optimum conditioning periods. To arrive at the optimum period for conditioning during step 1, one runs a series of tests, the first being taken immediately at the end of grinding and further tests at intervals of a few minutes up to an hour of conditioning subsequent to completion of grinding. If no economic improvement is shown by additional conditioning after grinding, one would, of course, immediately proceed with conditioning step 2 but if improvement is realized with further conditioning, the cutoff point in time will be a factor of the increased mineral recovery vs. the cost of further conditioning. This may be illustrated by comparing the results which were obtained with tests 150 and 162 which will now be described as examples II and III respectively.

EXAMPLE II

Test 150

The wetting agent, sodium carbonate, ammonium hydroxide and copper sulphate were added to the grinding circuit at the rates of 0.075, 7.5, 0.62 and 0.45 lbs. per ton respectively. Step I took place simultaneously with the grinding and step 2 was a 7 minute conditioning cycle with 0.125 lbs. per ton of potassium amyl xanthate (Z6) added. In step 3, 35 lbs. per ton of sodium silicate was added and the conditioning time was 10 minutes. The frother was pine oil and during rougher flotation 0.05 lbs. per ton of Z6 was added.

The rougher tailings were 80.1 percent by weight and analyzed 0.101 percent nickel.

EXAMPLE III

Test 162

This test is a near duplicate of test 150 with the exception that step 1 included a 45 minute conditioning period following the grinding stage. All other conditions were the same with the exception that slightly less wetting agent was used, namely 0.0625 lbs. per ton.

The rougher tailings were 79.8 percent by weight analyzing 0.097 percent nickel.

In comparing this test to test 150 the inclusion of additional conditioning following grinding and before the xanthate conditioning indicates a slightly higher recovery but may not be economically justified.

The optimum conditioning time for the second conditioning step is illustrated by table II below which describes five tests carried out under the same conditions except for the length of time devoted to conditioning step 2. As with all the tests reported throughout this specification, the work was done on three samples of the same copper nickel ore basically varying in talc content from an estimated minimum of 25 percent to a maximum of 50 percent. The series of tests making up each individual series were, of course, conducted upon the same sample of ore.

TABLE II

Text No.	Conditioning time, mins.			Rougher tailings		
	Step 1*	Step 2	Step 3	Percent wt.	Percent Ni	Percent Cu
200.....	Grinding cycle only through-out tests.	0	10	78.2	0.113	0.019
201.....		2.5	10	77.4	0.110	0.018
202.....		5.0	10	77.9	0.109	0.019
203.....		10.0	10	75.6	0.101	0.018
204.....		20.0	10	75.6	0.097	0.018

The major break in the tailings loss takes place between 5 and 10 minutes conditioning time and shows a further small in-

EXAMPLE IV

In the following comparative tests 120 and 123, all reagents and conditioning periods were the same with the exception that in test 120, 37½ pounds per ton of sodium silicate was used as against 30 pounds of sodium silicate per ton of ore in test 123. It was found that in test 123 the sodium silicate concentration had dropped below the lower effective concentration of the dispersant for optimum metallurgical results. In test 120, the percentage weight of the ore rejected in the tailings was 78.1, analyzing 0.130 percent nickel, as against the lower tailings rejection in test 123, that is 74.7 percent by weight analyzing 0.144 percent nickel. It is obvious that the percentage of material floating suffered and that there was a higher nickel loss in the tailings when the sodium silicate concentration is lower than that required to effectively disperse and depress the host rock minerals.

While I generally prefer to add all the dispersant during the third-conditioning step, I have found it possible to add a minor part in step 1 and the major part in step 3. Results obtained from adding a portion of the dispersant during this first step are shown in table IV.

TABLE IV

Test No.	Na ₂ SiO ₃ , lbs./ton		Conditioning time, mins.			Rougher tailings		
	Step 1	Step 3	Step 1*	Step 2	Step 3	Percent wt.	Percent Ni	Percent Cu
204.....	0	35	Grinding cycle only through-out tests.	20	10	75.6	0.097	0.018
209.....	10	25		20	10	74.6	0.098	0.018
217.....	17.5	17.5		20	10	75.0	0.103	0.019

crease in recovery at 20 minutes. Optimum condition time for step 2 is seen to be between about 10 to 30 minutes.

The time period of the third-conditioning step is similarly predetermined by experimentation and a group of such experiments are illustrated in table III describing four tests in which the only variable is the period allotted to the third-conditioning step.

TABLE III

Text No.	Conditioning time, mins.			Rougher tailings		
	Step 1*	Step 2	Step 3	Percent wt.	Percent Ni	Percent Cu
210.....	Grinding cycle only through-out tests.	20	2.5	74.6	0.096	0.018
211.....		20	5.0	75.6	0.097	0.017
204.....		20	10.0	75.6	0.097	0.018
212.....		20	20.0	75.7	0.103	0.018

It will be noted that the tailings loss increases between 10 and 20 minutes conditioning with no noteworthy differences in metallurgy between 2½ and 10 minutes. It may be seen from this table that the third-conditioning step for optimum results should not be carried on for more than 10 minutes and the preferred period is some 2½ to 5 minutes. It will also be noted that at 20 minutes metallurgy suffers. In this cycle it is therefore important not to exceed the selected time period by an appreciable time.

The amount of sodium silicate which should be present during the third conditioning step must also, in the case of each ore, be predetermined by experiments. On an ore of this type with high talcose content using conventional flotation means well known to the art, the mass of the ore tends to float producing a very low grade concentrate. To obtain the optimum amount of sodium silicate required to substantially disperse and depress the host rock minerals, I keep increasing the amount of sodium silicate until a comparatively clean differential sulphide float is obtained. I then continue with a slight excess over this amount and then conduct a series of controlled tests using lesser amounts until the host rock again begins to interfere with the float and the amount of sulphide mineral in the tailings increases. The optimum range of sodium silicate concentrate is now indicated. The following are two comparative tests showing the lower end of the range compared to the optimum concentration of sodium silicate which is approximately 25 percent higher than the lower limit tested.

*In tables II, III and IV the step 1 conditioning cycle coincides with the time of grinding. No additional conditioning has been done subsequent to grinding.

In comparing the results of these tests to 204 wherein all of the dispersant was added to step 3, as against approximately 30 percent to the grinding circuit with 70 percent to step 3 (test 209), the results, although slightly poorer, may be considered a standoff.

With 50 percent (test 217) of the dispersant added to the grinding circuit and 50 percent to step 3, there is a noticeable increase in tailings loss of nickel. From an operational point of view where it may be desirable to circulate part of the recovered solution from the flotation circuit tailings to the grinding circuit, and wherein there will be a number of the reagents including sodium silicate partially recovered, this characteristic of the circuit in being able to tolerate somewhat in excess of 30 percent of the total sodium silicate required without undue nickel loss can be important economically in reduction in the overall reagent cost as shown in this open circuit work.

The effect of reversing the times of addition of the sodium silicate dispersant and the xanthate collector is illustrated in table V in which the sodium silicate is added during step 2, and the xanthate in step 3.

TABLE V

Test No.	Conditioning time, mins.			Rougher tailings		
	Step 1	Step 2	Step 3	Percent wt.	Percent Ni	Percent Cu
205.....	0	10	5.0	76.9	0.116	0.019
206.....	0	10	10.0	76.3	0.114	0.019
207.....	0	10	20.0	75.7	0.108	0.018

When the results of these three tests are compared with the results obtained in test 204 as set out in table III, it will immediately be apparent that this reversal of the relative times of addition shows increased nickel losses although there is still considerable improvement over any prior knowledge in the art in the treatment of such talcy-natured ores.

EXAMPLE VI

This example, which includes a description of rougher concentrate cleaning, was carried out on an ore sample taken from the same ore body as the samples used in the preceding examples. This sample, however, had a higher percentage of

amount where further quantities show no further beneficial effect

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3. A process as defined in claim 1, in which the first-conditioning step is at least partially carried out during one or more wet-grinding stages.

4. A process as defined in claim 1, in which the entire first-conditioning step is carried out subsequently to one or more wet-grinding stages.

5. A process as defined in claim 1, in which the alkaline agent is sodium carbonate.

6. A process as defined in claim 1, in which the alkaline agent is ammonium hydroxide.

7. A process as defined in claim 1, in which the alkaline agents are sodium carbonate and ammonium hydroxide.

15 8. A process as defined in claim 1, in which the alkaline agents are sodium carbonate, ammonium hydroxide and calci-

um hydroxide.

9. A process as defined in claim 1, in which the alkaline agents are sodium carbonate and sodium hydroxide.

20 10. A process as defined in claim 1, in which the alkaline agents are sodium carbonate and calcium hydroxide.

11. A process as defined in claim 1, in which copper sulphate is added as an activating agent during said first-conditioning step.

25 12. A process as defined in claim 1, in which a trimethyl nonyl ether of polyethylene glycol is added as a wetting agent during said first conditioning step.

13. A process as defined in claim 1, in which the xanthate reagent employed is sodium amyl xanthate.

14. A process as defined in claim 1, in which the xanthate reagent employed is potassium amyl xanthate.

15. A process for the flotation recovery of copper and nickel sulphide minerals from ores containing both such minerals by a process in which at least two-conditioning steps

35 precede flotation, said process consisting essentially of (a) a first-conditioning step wherein the ore, together with such ac-

activating agents and wetting agents as are to be used, is conditioned in the presence of at least one alkaline agent selected from the group consisting of alkali metal carbonates, ammoni-

um hydroxide, mixtures of these two compounds, and mixtures comprising at least one of these compounds and at least one hydroxide selected from the group consisting of sodium

potassium, and calcium hydroxides, in an amount calculated to bring the pH of the resulting pulp up to a point within the optimum range of flotation of the said minerals, for a period of

time sufficient to activate said minerals, then (b) a second-conditioning step wherein the pulp resulting from the first step is conditioned in the presence of a selected member of the

family of xanthate flotation reagents for a period of time
50 determined by experiment to produce maximum activation of
said minerals; then (c) a third conditioning step wherein the

pulp resulting from the second step is conditioned in the presence of sodium silicate in an amount and for a time, predetermined by experiment, to be sufficient to effectively

55 disperse the host rock minerals; and then (d) subjecting the resulting pulp to froth flotation.

16. A process as defined in claim 15, in which the second-conditioning step is carried out for a period of from 10 to about 30 minutes.

60 17. A process as defined in claim 15, in which the third-conditioning step is carried out for a period of less than 10 minutes.

19. A process as defined in claim 15, in which the sodium silicate is added as dispersant during both the first and third-conditioning steps, the amount added in the first step being less than that added in the third step.