Patented June 20, 1933

1,914,694

UNITED STATES PATENT OFFICE

LAWRENCE H. LANGE, OF SALT LAKE CITY, UTAH, ASSIGNOR TO THE GENERAL ENGINEERING COMPANY, OF SALT LAKE CITY, UTAH, A CORPORATION OF UTAH

CONCENTRATION OF PHOSPHATE-BEARING MATERIAL

No Drawing. Application filed February 4, 1931. Serial No. 513,353.

This invention relates to the concentration of phosphate-bearing material and has for its object certain improvements in the method of concentrating phosphate-bearing material by froth flotation. The invention relates more particularly to the froth flotation of phosphatic material associated with quartz gangue constituents.

In the heretofore customary method of concentrating phosphate-bearing material by froth flotation, the phosphate-bearing particles are floated while the gangue constituents are depressed. The concentration of phosphate-bearing material in this manner is rendered somewhat difficult because the phosphate-bearing particles tend to partake of the nature of the gangue constituents. Although a substantial portion of the phosphate-bearing particles may be thus concentrated, there is a tendency for gangue constituents to accompany the phosphate particles into the froth layer maintained on the surface of the body of pulp undergoing the froth flotation operation, as well as for phosphate-bearing particles to be depressed together with gangue constituents that go into the tailings.

As a result of my investigations, I have determined that phosphate-bearing material may be subjected to a froth flotation operation in which the gangue constituents are floated and the phosphate-bearing particles are depressed. Flotation reagents may be employed that alone or in combination with one another selectively float the gangue constituents and selectively depress the phosphate-bearing particles. This "up-side-down" step is particularly effective in separating quartz constituents from phosphate-bearing particles.

According to the present invention, phosphate-bearing material is subjected in the form of a pulp to a froth flotation operation in the presence of flotation reagent adapted selectively to float gangue constituents and to depress phosphate-bearing particles.

While the flotation process of the invention may be conducted with phosphate-bearing material containing slimes, a better separation of the phosphate-bearing particles from the gangue constituents is obtained when the slimes are first substantially removed. The presence of slimes in the pulp is furthermore objectionable because the consumption of flotation reagents tends to increase.

In order to obtain a sharp separation of phosphate-bearing particles from gangue constituents, the material should be in a fine state of subdivision. As far as my investigations thus far have gone, best results appear to be obtained when the flotation operation is conducted on material ranging in size from approximately 28 to 200 mesh.

Phosphate-bearing material of the fine state of subdivision contemplated necessarily includes a substantial amount of minute particles falling within the so-called slime range. It is therefore better practice first to subject the phosphate-bearing material in the form of an aqueous pulp to a de-sliming operation. The slimes may be removed by a preliminary froth flotation treatment, tabling, classification or the like, before the addition of flotation reagents adapted to float the gangue constituents. Thus, any suitable frothing agent of commerce may be employed; such as pine oil, coal tar derivatives, wood-oils, or any appropriate hydrocarbon and the like.

The de-slimed phosphate-bearing pulp is then suitably treated in order to effect the froth flotation of gangue constituents and to depress phosphate-bearing particles. In order to inhibit the flotation of the phosphate-bearing particles, while leaving the gangue constituents in a condition amenable to flotation, flotation reagents functioning in a peculiar and novel manner must be employed.

Gangue constituents such as quartz are not readily floated in an acid circuit. It is therefore necessary to render the mineral pulp properly alkaline. Lime (CaO) is highly useful for this purpose. In fact, it serves a triple function. While assisting in rendering the pulp highly alkaline, it also acts as a depressant to the phosphate-bearing particles; and as a collector for the gangue particles when used in conjunction with another appropriate reagent, such as sodium oleate.

Other reagents, such as caustic soda, sodium carbonate and the like may also be employed to produce alkalinity; but their use appears to necessitate the employment of more depressant reagents than is needed when they are employed in conjunction with lime. These other alkaline reagents do not, moreover, appear to yield as marked a differential separation as with the use of lime.

In order to make the gangue constituents, such as quartz, floatable, suitable flotation reagents must be employed. Thus, compounds producing the oleate radical and metallic salts, such as lead nitrate, in solution, are highly effective. For example, a metallic soap such as sodium oleate in the form of red oil soap, acid mass sodium oleate, or ordinary soap are suitable. A soap compound called "oleate" on the market is particularly efficacious in the operation contemplated. Metallic salts such as ferrous sulphate, copper sulphate, zinc sulphate, aluminum sulphate, lead nitrate, copper nitrate, ferrous nitrate, and ferric nitrate or the like may be employed to assist in the flotation step. Alcoholic solutions (denatured alcohol or methanol) of the different soaps and oleic acid are highly effective in causing quartz to float.

As stated above, the presence of lime in the pulp body not only assists in making the circuit alkaline, but it also tends to exert a depressant action on the phosphate-bearing particles. This depressant action is materially aided by the addition of a proper amount of a suitable carbohydrate, such as the well known starches. Starch and lime in the mineral pulp not only tend to sink the phosphate-bearing particles, but they also assist in modifying the gangue constituents so as to make them amenable to the floating step.

Although the soaps produce considerable froth, the froth does not generally have sufficient stability to support its burden of gangue constituents. Additional frothing agent or agents should therefore be employed in conjunction with those particularly intended to depress the phosphate-bearing particles and to float the gangue constituents. Practically any of the conventional frothing agents may be employed, such as pine oil, coal tar derivatives, wood oils and the like. A coal tar derivative called "tarol" has been found to give unusually good results.

The flotation operation may be conducted in any suitable froth flotation apparatus. Thus, the well known Callow or MacIntosh cells may be employed. Finally disseminated air under pressure is released into the body of pulp at or near its bottom. As the air bubbles rise they tend selectively to be attached to the gangue constituents and lift them to the froth layer maintained on the surface.

The procedure for agitation of the pulp and flotation may be varied in certain respects. Thus, when employing the lime and starch, agitation should precede the flotation step. If tarol is used, it should be added during the agitation period in order to cause dispersion of the reagent throughout the pulp body. If, on the other hand, pine oils are used, they may be added either before or after agitation of the pulp body. The point at which the frothing agent is added depends largely on its property of dispersion in aqueous solutions.

The metallic salts, such as, for example, copper sulphate, may be added either before or after agitation; but when added before agitation, the soap consumption is decreased. The soaps have no marked effect when added to the pulp during the agitation period. Hence, it is preferable to add the soap to the pulp in the flotation cell, where its desired action may at once be obtained.

If tarol is employed as a frothing agent, sodium oleate is the only reagent that needs to be added to the alkaline flotation circuit. Excellent results are obtained when employing this combination. To obtain the greatest efficiency and consequently to decrease excess consumption, it is advantageous to add the soap at intervals throughout the duration of the flotation step.

When employing lime and tarol, they are preferably added to the mineral pulp during the agitation period; i. e. prior to the actual flotation of the quartz constituents. As a result of the agitation of the pulp in the presence of the lime and tarol, the slimes are substantially floated away. The oleate is then added, and the quartz particles are removed by froth flotation.

Extensive deposits of so-called phosphate rock are found in Florida. This phosphate-bearing material contains substantial amounts of quartz, sometimes referred to as silica sand. The material is found in nature for the most part in a finely subdivided state, many of the smallest particles falling within the so-called slime range. The phosphate is present generally as tricalcium phosphate. My invention as practiced on this material has shown very excellent results. To illustrate the practice of the present invention, the data submitted below may be considered in the examples set out.

The amounts of reagents specified are, as is conventional, computed on the basis of per ton of feed treated.

Example No. 1

In a test run on minus 28 to plus 48 mesh feed, the following procedure was followed:—the charge was given a five minute agitation (2 to 1 pulp density) with 15 pounds lime, ½ pound starch and No. 116 Yarmor pine oil. Flotation of the quartz was brought about with the addition of 3

pounds of acid mass sodium oleate (in denatured alcohol solution), 0.8 pound of lead nitrate, and 0.5 pound Yarmor pine oil. The flotation pulp density was 7 to 1.

Percent B. P. L. (bone phosphate of lime) in concentrates=15.2%.

Percent B. P. L. (bone phosphate of lime) in tailings=66.0%.

Recovery of B. P. L. in tailings=77.%.

Example No. 2

In a test run on minus 48 to plus 200 mesh feed, the following procedure was employed, producing a good grade of quartz concentrate with very small amounts present in the tailing:—an average portion of the ore was agitated for a period of five minutes at 1 to 1 pulp density with 25 pounds lime, 2 pounds starch, 1 pound copper sulphate, and 2 pounds tarol. After agitation, the slimes produced therefrom were floated off, followed by the addition of 1.35 pounds of sodium oleate (red oil soap). Flotation was carried on at a pulp density of 3 to 1.

Percent B. P. L. in concentrates=15.2%.
Percent of B. P. L. in tailings=71.83%.
Recovery of B. P. L. in tailings=70.%.

Example No. 3

In another test, an average portion of the ore was first de-slimed and then screened on a 48 mesh screen, the oversize particles being ground in a ball mill for 8 minutes; yielding a ground product containing 8% plus 48 mesh particles. The material is ground in order to reduce the size of the coarser particles of quartz to make them more amenable to flotation. The ground product was then roughly de-slimed, and the de-slimed product was added to the minus 48 mesh feed from the screening. This product was then given a 10 minute agitation (1 to 1 pulp density) with 25 pounds lime (CaO) per ton of ore and 3 pounds of tarol per ton of ore. After agitation, the slimes produced therefrom were floated off, followed by the addition of 1.0 pound oleate soap compound. The flotation operation was then carried out at a pulp density of 3 to 1.

	Percent
Percent B. P. L. in slimes from grinding	63.9
Percent B. P. L. in concentrates	12.4
Percent B. P. L. in tailings	73.9
Recovery of B. P. L. in slimes	7.3
Recovery of B. P. L. in tailings	65.4
Total recovery of B. P. L.	72.7

The rise of the gangue particles and the fall of the phosphate-bearing particles appear to be due in large part to the manner in which the pulp is initially conditioned. As pointed out above, the pulp is suitably conditioned with the alkaline agent in advance of the flotation step itself. The conditioning period, for example 30 minutes, with the lime appears to cause a wetting of the phosphate-bearing particles, and thus renders them non-amenable to froth flotation when the collecting agent, such as soap, is ultimately added. The frothing agent, such as steam distilled pine oil, is advantageously present in the pulp during the conditioning period, thus permitting a thorough dissemination of the pine oil prior to the introduction of the pulp to the flotation cell.

I claim:

1. In a method of concentrating phosphate-bearing material, the step which comprises subjecting the material in the form of an alkaline pulp to a froth flotation operation in the presence of sodium oleate and a suitable lead nitrate salt in solution adapted to float quartz gangue constituents.

2. In a method of concentrating phosphate-bearing material, the step which comprises subjecting the material in the form of an alkaline pulp to a froth flotation operation in the presence of sodium oleate and lead nitrate solution adapted to float quartz gangue constituents.

3. In a method of concentrating phosphate-bearing material, the step which comprises subjecting the material in the form of an alkaline pulp to a froth flotation operation in the presence of sodium oleate and lead nitrate adapted to float quartz gangue constituents and in the presence of an oil adapted to depress phosphate-bearing particles.

4. In a method of concentrating phosphate-bearing material by froth flotation, the step which comprises subjecting the phosphate-bearing material in the form of an alkaline pulp to a conditioning period until the phosphate-bearing particles are rendered non-amenable to froth flotation when a collecting agent is added to the pulp whereby quartz gangue constituents may be selectively floated while the phosphate-bearing particles are depressed.

5. Method according to claim 4, in which lime is added to render the pulp alkaline and to depress phosphate-bearing particles.

6. Method according to claim 4, in which a suitable hydrocarbon is added to make a stable froth and to depress phosphate-bearing particles.

7. Method according to claim 4, in which a suitable oil is added to make a stable froth and to depress phosphate-bearing particles.

8. Method according to claim 4, in which a suitable coal-tar derivative is added to depress phosphate-bearing particles.

9. Method according to claim 4, in which lime and a suitable oil are added to render the pulp alkaline and to depress phosphate-bearing particles.

10. Method according to claim 4, in which

lime and a suitable coal-tar derivative are added to render the pulp alkaline and to depress the phosphate-bearing particles.

11. Method according to claim 4, in which a soap is added to float quartz gangue constituents.

12. Method according to claim 4, in which sodium oleate is added to float quartz gangue constituents.

13. Method according to claim 4, in which acid mass sodium oleate is added in alcoholic solution to float quartz gangue constituents.

14. Method according to claim 4, in which a metallic salt in solution is added to float quartz gangue constituents.

15. Method according to claim 4, in which a soap and a metallic salt are added in solution to float quartz gangue constituents.

16. Method according to claim 4, in which lime, a soap and a suitable oil are added.

17. Method according to claim 4, in which lime, sodium oleate and a suitable oil are added.

18. Method according to claim 4, in which lime, a soap and a suitable coal-tar derivative are added.

19. Method according to claim 4, in which lime, a soap, a metallic salt in solution, and a frothing oil are added.

20. Method according to claim 4, in which the pulp of phosphate-bearing material is initially subjected to a slime removal operation.

21. Method according to claim 4, in which the conditioning period is effected in the simultaneous presence of lime and a frothing oil.

22. Method according to claim 4, in which the conditioning period is effected in the simultaneous presence of lime and a frothing oil, and then subjecting the pulp so treated to a froth flotation operation in the presence of soap.

In testimony whereof I affix my signature.

LAWRENCE H. LANGE.