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CONCENTRATION OF PHOSPHATE BEARING MATERIAL

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This invention relates to concentration of ores and is herein described as applied to the concentration of a so-called pebble phosphate ore of Florida. In this ore much of the gangue is silica or quartz.

These ores are deslimed and are screened to separate out the coarser material over 3/32 inch or 3/64 inch, which is rich, usually rich enough to ship to fertilizer factories. This desliming is often facilitated by pumping a thin pulp of the ore through long pipes to the concentrating plant, this also to some extent dislodging particles of slime normally adherent to the phosphate particles. The deslimed material is classified as to size of particles, and then frequently the finer material resulting from this classification is concentrated by subjecting it to a flotation operation, while the coarser material resulting from this classification is subjected to a form of table concentration. Frequently such an ore can be economically handled without subjecting any portion of the deslimed and screened material to a grinding operation, with the result that none of the recoverable phosphate is lost by being ground, incidentally or otherwise, to a slime. The economies thus obtained arise in two ways. First, as mentioned above, no new unrecoverable slime is created, and, secondly, no excessive amount of added agents is needed to make up the extra reagents required when very fine slime material is being concentrated.

The same agents are usually employed both for flotation concentration and for table concentration. This arises from the nature of the table concentration employed in which the phosphate comes off the side of the table as if it were the lighter material while the gangue comes off the end of the table.

According to the present invention there is utilized as the novel agent a water-insoluble soap, preferably made in situ, with added inorganic soluble substances, and it is found that the process works best in an alkaline pulp. The alkalinity found most useful often shows a pH value of between 8. and 9.6. At this degree of alkalinity it is often found that the flotation of the valuable phosphate is most rapid while there is less than the normal tendency to float the gangue, which is principally quartz.

Lime, in amounts to produce this pH value, has proved the most useful water-insoluble soap-producing substance both for richness of concentrate and for recovery of phosphate by flotation, but other substances have also proved useful, even though they produce a different pH value in the pulp which is being concentrated. It is believed that the insoluble soap which is formed to some extent coats the surface of the phosphate particles.

The process was carried out in the laboratory by grinding the phosphate-bearing material to pass twenty-mesh or in some instances thirty-five mesh, agitating the ground material with enough water to enable water to be decanted and carry off with it suspended slimes, and repeating decantation two or three times. The resulting deslimed pulp, containing 30% to 60% water was treated with caustic soda, added in solution in an amount equal to 0.40 pounds per ton, dry weight, of the material. Then there was added fatty acid, preferably in solution in an unsaponifiable oil such as fuel oil, during agitation of the thick pulp. Various fatty acids may be used, but oleic acid is a convenient standard for tests. Thereafter more fuel oil was added to bring its total amount up to two pounds per ton of dry material, and there was also added a mineral-frothing agent, such as Hercules Powder Company's Tarol #2 (a mixture of 75% steam distilled pine oil and 25% rosin oil by volume), in amount equal to 0.14 pounds per ton of dry material.

The thick pulp was then diluted with water carrying in solution the inorganic substance, such as caustic lime, which reacts with fatty acid to form insoluble soap; and was immediately agitated to yield the desired float concentrate. When caustic lime was thus added in solution it was usually found that half a pound of lime per ton of dry material yielded the desired pH value. The rougher concentrate obtained by one

agitation may be reagitated to yield a cleaner concentrate and a middling.

Tests were also made with substitutes for caustic lime, such as calcium chloride, $CaCl_2$, calcium nitrate $Ca(NO_3)_2$, ammonia alum, $Al_2(SO_4)_3 (NH_4)_2 SO_4.24H_2O$, and crystalline zinc sulphate.

The following table shows the comparative results obtained with various amounts of oleic acid (Column 1), with the various inorganic compounds (Column 2), in the number of seconds of agitation and separation by subaeration, indicated as time of flotation (Column 3). The inorganic substance, used in all except the first test was added at the rate of half a pound per ton of dry ore treated. Column 4 gives the assay in bone phosphate of lime of the raw material of each test, calculated from the assay of the products obtained. Column 5 gives the assay of the finished or cleaner concentrate obtained by retreatment of the rougher concentrate. Column 6 gives the assay of the combined middlings and tailings. In plant practice the middlings are returned for further treatment. Column 7 gives the percentage of original bone phosphate of lime recovered in the concentrate. Column 8 gives the ratio of concentration.

Test no.	Oleic acid (red oil)	Agents	Flotation time seconds	Assays per cent		B. P. L. per cent		Ratio conc.
				Head	Concs.	Midd. & tail	B. P. L. rec.	
1	0.32	None	60	22.96	72.91	5.08	90.9	2.43
2	0.32	$CaCl_2$	45	22.91	73.21	5.89	89.9	2.47
3	0.32	$Ca(OH)_2$	45	23.03	74.34	3.86	83.5	2.70
4	0.42	$Ca(OH)_2$	45	22.91	73.21	5.03	91.0	2.44
5	0.32	$Ca(NO_3)_2$	45	22.69	72.72	4.75	91.4	2.43
6	0.32	$Al_2(SO_4)_3.(NH_4)_2SO_4.24H_2O$	60	22.85	72.30	4.80	91.3	2.43
7	0.32	$ZnSO_4.6H_2O$	50	22.85	71.50	3.68	83.6	2.33
8	0.26	$ZnSO_4.6H_2O$	45	22.83	71.98	4.71	91.7	2.39
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)

Test 1 was a blank test under standard conditions as described above without the addition of any inorganic substance, for comparison with the results respectively obtained by the addition of inorganic substances.

It will be noted that with shorter periods of agitation the tests including the added inorganic substances, except alum, gave either richer concentrates or larger recoveries, and that alum for the same time of agitation gave a larger recovery.

The above examples showing comparative results in flotation operations will be substantially paralleled in table concentration operations employing the same reagents. In commercial operations, as usual, treatment will vary slightly with the ore used and with the water used. The presence of insoluble soap in the commercial concentration of phosphate-bearing material has been found especially useful when the pulp which is to be concentrated contains an unusually large proportion of fine silica. This fine silica gangue tends to float with the valuable phosphate concentrate, with the result that low grade concentrates are produced.

It has been found that the addition to the pulp of about half a pound of lime per ton of dry material is a very economical method of substantially diminishing flotation of the fine silica. It has been found best to add the lime after any alkali or soap or oil or other flotation agents.

The lime has been added with satisfactory results to the feed of the minerals separation flotation machines used for the recleaning operation in the process shown in the patent to Littleford 1,780,022, dated October 28, 1930. In an alternative procedure, wherein a minerals separation flotation machine was used to produce the rougher concentrate, it was found advantageous to add the lime in the make-up water used for thinning the pulp just before it entered this machine, but after the alkali and other flotation agents had been added.

It has also been found that lime, added as described in commercial operations often changes a flotation operation from one yielding a sluggishly rising float to one in which the float rises rapidly, appears to be more buoyant, and carries increased phosphate values. Thus addition of the lime sometimes causes more of the float to rise on the head flotation cells of a flotation machine thus leaving less valuable material to be floated in the final flotation cells.

Having thus described certain embodiments of the invention, what is claimed is:

1. The process of concentrating a phosphate-bearing material which consists in agitating a pulp of the phosphate-bearing material with a fatty acid, then agitating with a material which reacts with the fatty-acid to form an insoluble soap and separating a concentrate.

2. The process of concentrating a phosphate-bearing material which consists in agitating a pulp of the phosphate-bearing material with a fatty acid and an unsaponifiable oil, then agitating with a material which reacts with the fatty acid to form an insoluble soap and separating a concentrate.

3. The process of concentrating a phosphate-bearing material which consists in treating a pulp of the material with alkali,

then agitating with a fatty acid, then agitating with a material which reacts with the fatty acid to form an insoluble soap and separating a concentrate.

4. The process of concentrating a phosphate-bearing material which consists in agitating a thick pulp of the material with a fraction of a pound of caustic alkali per ton of dry material, then agitating with a fraction of a pound of fatty acid per ton of dry material, then further agitating in the presence of unsaponifiable oil, then agitating the thinned pulp with the further addition of a fraction of a pound per ton of dry phosphate-bearing material of a material which reacts with the fatty acid to form an insoluble soap and separating a concentrate.

5. The process of concentrating by flotation a phosphate-bearing material which consists in agitating a thick pulp of the material with a fraction of a pound of caustic alkali per ton of dry material, then agitating with a fraction of a pound of fatty acid per ton of dry material, then further agitating in the presence of unsaponifiable oil, then agitating the thinned pulp with the addition of a fraction of a pound per ton of dry phosphate-bearing material of a material which reacts with the fatty acid to form an insoluble soap, the last mentioned agitation forming a float, and collecting the float.

6. The process of concentrating a phosphate-bearing material which consists in agitating a thick pulp of the material with a fraction of a pound of caustic alkali per ton of dry material, then agitating with a fraction of a pound of fatty acid per ton of dry material, then further agitating in the presence of unsaponifiable oil and also in the presence of a mineral-frothing agent, then agitating the thinned pulp with the further addition of a fraction of a pound per ton of dry phosphate-bearing material, of a material which reacts with the fatty acid to form an insoluble soap and separating a concentrate.

7. The process of concentrating a phosphate-bearing material which consists in treating a thick pulp of the material so as to agitate the material with soap and unsaponifiable oil, thinning the pulp with water carrying in solution an insoluble-soap-forming material and separating a concentrate.

8. The process of concentrating a phosphate-bearing material which consists in treating a thick pulp of the material so as to agitate the material with soap and unsaponifiable oil, thinning the pulp with water carrying lime in solution so as to produce a pulp having a pH value in the neighborhood of 9, and agitating the pulp and separating a concentrate.

9. The process of concentrating a phosphate-bearing material which consists in treating a thick pulp of the material so as to agitate the material with an amount of soap in the neighborhood of a pound per ton of the dry phosphate-bearing material, also agitating with several times that amount of insoluble oil and also with a mineral-frothing agent, thinning the pulp with water carrying such amount of lime in solution as to produce a pulp carrying about half a pound of lime (CaO) per ton of the phosphate-bearing material, agitating the pulp and separating a concentrate.

10. The process of concentrating a phosphate-bearing material which consists in treating a thick pulp of the material so as to agitate the material with an amount of soap in the neighborhood of a pound per ton of the dry phosphate-bearing material, also agitating with several times that amount of insoluble oil and also with a mineral-frothing agent, thinning the pulp with water carrying such amount of zinc salt in solution as to produce a pulp carrying about half a pound of a zinc salt per ton of the phosphate-bearing material, agitating the pulp and separating a concentrate.

11. The process of concentrating a phosphate-bearing material which consists in treating a thick pulp of the material so as to agitate the material with an amount of soap in the neighborhood of a pound per ton of the dry phosphate-bearing material, also agitating with several times that amount of insoluble oil and also with a mineral-frothing agent, thinning the pulp with water carrying such amount of aluminum salt in solution as to produce a pulp carrying about half a pound of the aluminum salt per ton of the phosphate-bearing material, agitating the pulp and separating a concentrate.

In testimony whereof, we have signed this specification.

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