

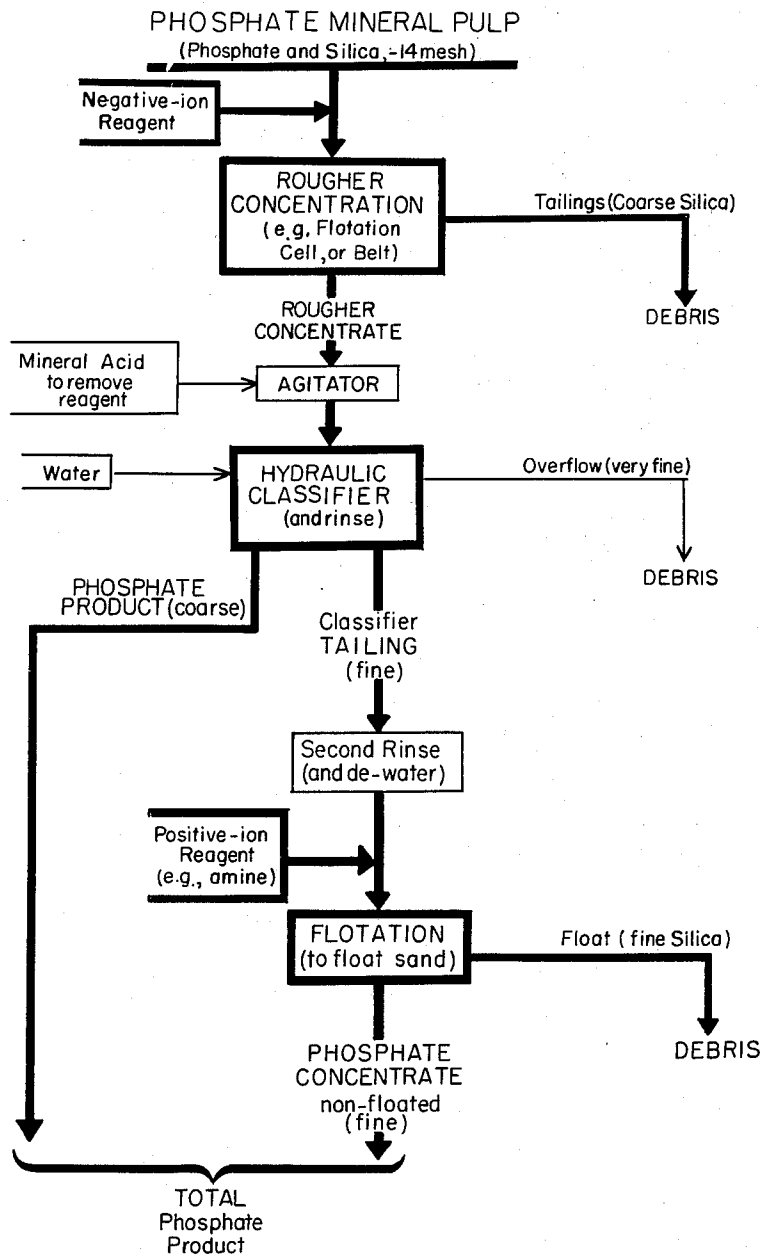
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CONCENTRATION OF PHOSPHATE MINERAL

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CONCENTRATION OF PHOSPHATE MINERAL

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This invention relates to the concentration of phosphate mineral and particularly to operations for the ultimate recovery of high grade phosphate material from phosphate ore or from parts or fractions of such ore, by wet concentration procedure. While to a considerable extent valuable phosphate of satisfactory grade can be separated from suitable phosphate rock or ore by so-called washing or other relatively simple classifying procedure, especially with respect to the larger particle sizes, the remaining fractions of such ores, or other phosphate mineral materials, must usually be subjected to one or more concentrating operations, with appropriately selected reagents or the like, in order to derive a product having a suitably high content of phosphate, relative to the siliceous gangue, commonly present in or with the ore as mined. The present invention is designed to afford a more effective and especially more economical method of treating such phosphate mineral to yield a final product or collection of concentrates having a satisfactorily high content of phosphate, e.g. a grade of at least about 70% B.P.L. (bone phosphate of lime) or better. While the improved procedure is applicable to phosphate mineral from various sources, and in all cases is of special advantage for mineral having the general characteristics of particle size and composition described below, it is exemplified herein as practised in the treatment of Florida phosphate, particularly certain fractions of such phosphate ore as remain after various washing or primary classifying operations for first separating a high grade phosphate of the larger particle sizes.

For example, after certain such preliminary operations, a mineral fraction is produced, having a particle size less than 14 mesh and usually —20 mesh, with a grade of only about 30% to 40% B.P.L. or thereabout. This material may have a considerable variety of particle size, with a preponderance between 28 mesh and 100 mesh, including a considerable part less than 48 mesh and some small part less than 100 mesh. Such mineral thus contains both coarse and fine particles of phosphate and both coarse and fine particles of impurity, i.e. siliceous material which can be generally or briefly designated as silica. It will be understood that the particle size designations relate to standard screen sizes, plus references meaning particles larger than the stated screen and minus references meaning particles that pass through the given screen. Hereofore one reasonably effective procedure for concentrating phosphate mineral of these characteristics has involved a combination of steps, first a flotation concentrating operation using so-called negative-ion reagent material, whereby a phosphate-enriched product is obtained, i.e. as the floated or elevated material in the flotation cells, with a tailing or non-floated discharge of siliceous material. This operation is designated as a rougher or roughing concentration, the discarded tailings being predominantly the larger particles of siliceous material, and the rougher concentrate containing most of the phosphate, including both large and small particle sizes, but also considerable siliceous gangue, especially in the small particle sizes. The

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rougher concentrate is then treated with mineral acid and subjected to a plurality of rinsing operations to remove the negative-ion reagents. It is then subjected to a second or finishing flotation concentration (which may actually require a plurality of steps) employing positive-ion reagents, such as amines, whereby a silica float is obtained and the final cell product or discharge, i.e. non-elevated material, is a phosphate concentrate of fairly good grade. Thus the froth product of the finishing concentration removes the finer particles of siliceous gangue, so that as far as practical, all sizes of phosphate appear in the ultimate, desired concentrate.

While this procedure is reasonably effective, it has some disadvantages, particularly in matter of expense, including the amount of equipment required, especially for all of the flotation operations, and also particularly in the cost of the relatively large quantities of reagent material needed for the finishing or sand-floating concentration stages. Some proposals have been made for modifying the above complete process to reduce its cost, but in general these have involved additional flotation steps and have fallen short of providing a conveniently practical solution to the problem. The present invention comprises a new and effective combination of steps for concentrating phosphate mineral, especially material of the character described above, whereby various substantial advantages are realized, in cost of reagents, in requirements as to equipment, and in other respects as explained below. Specifically the procedure involves first subjecting the mineral to a rougher concentration with negative-ion reagent material, to yield a rougher concentrate of phosphate-enriched nature, and then after treatment with mineral acid or the like, subjecting the rougher concentrate to hydraulic classification, whereby the material is segregated into fractions of different settling properties. According to a specific discovery of the invention, the larger size fraction, i.e. the faster-settling rate material, derived from the hydraulic classification, constitutes in itself a high grade phosphate product. The tailings from the hydraulic classification are thereafter subjected to froth flotation with positive-ion reagent material, to separate a silica float and thereby yield a further quantity of high grade phosphate product. The hydraulic classifying stage not only provides direct recovery of a portion of the phosphate of suitable quality but also performs the important function of rinsing the acid-treated material, in preparation for the ultimate positive-ion concentration operation on the finer or slower-settling fraction.

A variety of important advantages can be obtained with this process. In the first place, as indicated, the hydraulic classifying stage here involves the use of considerable supplemental or so-called hydraulic water for effectuating the desired separation of the particles according to settling characteristics (chiefly particle size but in some measure, specific gravity) and this step therefore serves also as an effective rinsing operation for carrying away the spent negative-ion reagents and the acid added to remove them. Since the sized product from the hydraulic classifier represents a considerable part, say from 10% to 50% or more (by weight) of the rougher concentrate, the amount of further equipment needed, especially in flotation cells for the final silica separation, is correspondingly reduced. Thus there is a definite saving in capital investment and in upkeep and operating attention to equipment.

A particularly important result of the described sequence of steps is the reduction in amount of reagent required for the silica flotation. Since the hydraulic classifier removes a considerable part of the rougher concentrate, the quantity of ore pulp going to the silica flotation cells is correspondingly reduced, this in itself providing a substantial saving of the relatively costly

amine reagent. Furthermore, the hydraulic classification removes most of the coarser phosphate particles, which in the prior procedure appear to break up in the sand flotation step, creating a considerable amount of extreme fines or slimes and necessitating the use of rather large amounts of the amine material in order to achieve the desired results. That is to say, the saving of reagent appears to be even greater than is accounted for by the reduction in the tonnage to the silica flotation cells, presumably by elimination (in the hydraulic classifier) of material which tended to waste this valuable reagent.

The procedure is found to afford a definite metallurgical advantage, i.e. in the ultimate recovery of a larger quantity of the phosphate originally present in the feed to the rougher stage. That is to say, taking the two product fractions together, i.e. the coarse or faster-settling material from the classifier and the non-floated product from the sand-flotation cells, there is an improvement in phosphate recovery. It is believed that this improvement stems particularly from the use of the hydraulic classifier, in that the high grade phosphate (faster-settling) product from such step appears to contain a larger portion of phosphate particles in its finer fractions than would be expected from a simple sizing operation such as by screening. This fact has been indicated by screen and chemical analyses of the materials at various stages in the process and is understood to arise because of slight difference in specific gravity between silica and phosphate particles. In other words, it appears that an improved recovery of phosphate is obtained because of special efficiency of the hydraulic classifying step, i.e. in comparison with the efficiency of the ultimate sand-flotation. The hydraulic classification used for the combined effects of sizing and rinsing is also found to require little or no attention from operating personnel, especially in using apparatus of the type now preferred for this stage, as explained below.

In general, the effectiveness of the improved process is predicated on the combined or cooperative result of the successive operations, i.e. in the correlation of the various types and sizes of particles removed or segregated in each stage. Specifically, by the rougher concentration, the coarse silica particles are essentially separated, the rougher concentrate thus consisting chiefly of the coarse and fine phosphate and the fine siliceous material. Instead of subjecting the entirety of such concentrate to the amine-reagent operation for floating the silica, the hydraulic classifying step in effect separates the coarse phosphate particles to provide, directly, a part of the ultimate, desired product, while essentially only the fine particles of both phosphate and silica remain in the tailing from the classifier. Thus the fine silica sand is thereafter separated in the final flotation stage, which involves a feed of reduced volume, containing substantially only the finer fraction of the original mineral material.

The nature of the specific steps in the present process may permit of some variation. For instance, the rougher concentration is readily effected by flotation, using negative-ion reagent material, e.g. fatty acid, with accompanying reagents of conventional character for such purpose, such as caustic and fuel oil. As will be understood, the floated product is the phosphate-enriched concentrate, the tailings or non-floated discharge constituting the coarse siliceous gangue, carrying relatively little phosphate. Alternatively, other apparatus or procedures may be employed for such rougher concentration, i.e. among various types of concentrating or like devices known to be useful to separate phosphate from silica with negative-ion reagents causing the phosphate to rise away from the gangue. For example, another type of such operation, which may be effectively used in the present procedure, is so-called belt concentration, where after being treated with reagents of the same character as described above (e.g. fatty acid, caustic and fuel

oil), the feed pulp is applied at the end of a long traveling belt, which receives further quantities of water along its length and which passes under a multitudinous series of collecting members, with the result that the material segregates, the phosphate being carried over the side of the belt and the coarse silica passing along and being dumped at the end as a phosphate-depleted tailing. The rougher product thus derived from the sides of the concentrating belts is found to have characteristics similar to those of the rougher flotation described above, namely embracing both coarse and fine phosphate particles and fine siliceous particles. Whereas in at least one conventional plant, the rougher concentrate is then taken to a further belt concentrator serving as a cleaner operation, the process of the present invention embraces the successive classifying and sand-floating stages, with considerable advantage in many cases, as in efficiency of overall recovery, quality of product, convenience of operation, and the like.

In the hydraulic classifying stage it is at present greatly preferred to employ apparatus involving a combination of hindered-settling and free-settling, such as disclosed in U.S. Patent No. 2,708,517, granted May 17, 1955, to R. D. Evans, with an automatically controlled siphon discharge for removing the faster-settling product from the foot of the teeter column, as disclosed in U.S. Patent No. 2,714,958, granted August 9, 1955, to R. D. Evans, another embodiment of such hydraulic classifying apparatus including the two types of settling and the siphon discharge being shown in U.S. Patent No. 2,784,841, granted March 12, 1957, to R. D. Evans. For convenience, such double-column hydraulic classifier (including the described siphon discharge) will be identified herein as the Evans hydraulic sizer. Such apparatus comprises an upper, open tank of upright cylindrical shape and relatively large diameter, having a frusto-conical bottom opening at its lower end into a vertical tank section of reduced diameter constituting the hindered-settling column.

An inverted, i.e. open bottom tank structure, is enclosed and spaced within the upper tank, with a discharge pipe from the top of such upper section, below the level of liquid in the main or large upper tank. This upper section constitutes a so-called upper column having its lower opening or mouth spaced above the top of the hindered-settling column. Hydraulic water is supplied through a constriction plate or the like at the foot of the hindered-settling column and the described siphon discharge is arranged to carry the classified or coarser product away from the foot of the last-mentioned column, i.e. just above the constriction plate. The feed pulp, which in the present process is the rougher concentrate, is supplied to the open top of the sizer, while hydraulic water is supplied at appropriate rate to the foot of the teeter or hindered-settling column. The material settles through the main or large tank to the space between the upper and lower columns; hindered-settling occurs in the lower column, while the finer fraction, in effect rejected for hindered-settling, is carried upward by the water flow through the upper column and is removed through its discharge pipe. The sharpness of classification is aided by the free-settling in the upper column where the larger or faster-settling particles may fall down to the vicinity of the lower or hindered-settling region, while the coarse product is derived from the siphon discharge through the bottom of the latter region. Excess water, together with extreme fines or slimes, is removed at an overflow of the main tank around the upper column.

The Evans classifier is unusually efficient and provides an exceptionally sharp segregation of the feed by settling characteristics (largely particle size) and its use in the present process is thus a special feature of invention, but other hydraulic classifiers or classifying operations may alternatively be employed, i.e. any of a variety of wet classifying devices which function by permitting or pro-

moting settling of the particles supplied and which may be defined as involving the use of considerable water additional to that of the feed pulp. In general, such additional water is introduced so that its direction of flow opposes that of the settling particles. Such hydraulic classifiers may be of either free-settling or hindered-settling type, or of combinations or modifications of such modes of operation, but in all cases functioning to produce a relatively rapid and relatively sharp classification of the supplied particles in accordance with settling rate. As employed in the present invention, hydraulic classification functions essentially by sizing, since the phosphate and gangue particles do not differ very greatly in specific gravity, but there is some segregation governed by the specific gravity difference, which favors the concentration of phosphate in the product as explained above. At the same time, and by reason of the hydraulic water added in substantial quantity and continuous flow for the functioning of hydraulic classification, the above-mentioned rinsing action is also achieved.

The accompanying drawing is a flow sheet of the process of the invention, as carried out in a presently preferred way.

The procedure can be more specifically described in connection with examples of its use as applied to particular kinds of mineral phosphate. Thus as a first example, the method has been utilized for treatment of a phosphate ore pulp in a phosphate washer plant that was supplied with phosphate rock or ore mined in the vicinity of Boyette, Florida. In this particular operation, the ore as mined had been subjected to certain preliminary and primary operations involving washing or screening and hydraulic classification, whereby phosphate of relatively large sizes had already been separated. The feed pulp for the present process, subjected to preliminary thickening to the extent necessary, represented the tailing or tailings from one or more hydraulic classifying operations, and constituted a moderately finely divided phosphate mineral or phosphatic sand, having a grade of about 30% B.P.L. (or sometimes as low as 20% B.P.L.) and having a particle size of -14 mesh, or more usually -20 mesh. Both the phosphate and the siliceous components of the material were present in a rather wide range of particle sizes, and there was a substantial content of both components throughout the range, although the phosphate grade of the larger sizes appeared considerably better. Specifically, the range extended from -20 mesh to a few percent -100 mesh, the greatly major part of the pulp being distributed through a size range from -28 mesh to +100 mesh. This pulp, in water, was treated with negative-ion reagent material comprising fatty acid and coating reagents. Specifically, a mixture of tall oil (a fatty acid-containing product), fuel oil and caustic was employed, the application of these reagents to the pulp being effected in a conditioner, where the pulp was agitated with the reagents, in accordance with conventional practice.

From the conditioner, the oiled pulp, i.e. carrying the negative-ion reagents, was fed to flotation cells, specifically so-called Airflow machines, operated in the conventional manner, to yield a phosphate concentrate and a tailing. This was the rougher operation, the floated product being the rougher concentrate which had a grade of about 65% B.P.L., while the tailing, consisting chiefly of the larger size siliceous gangue particles had a content of less than 15% B.P.L. This tailing was discarded, i.e. pumped to the debris repository of the plant.

The rougher concentrate was then treated with mineral acid, specifically sulfuric acid (although other mineral acid, such as hydrochloric, may be employed) for de-oiling, i.e. for removing the negative-ion reagents. This addition of acid was effected in an agitator, of conventional character for such purpose. From the acid agitator, the pulp was supplied continuously (all of the operations being continuous) into the open top of an Evans hy-

draulic sizer, as described above. This hydraulic classifier was operated with a continuing and relatively large supply of water through the constriction plate at the foot of the hindered-settling column, i.e. so-called hydraulic water. The withdrawal of the product from the foot of the hindered-settling column was adjusted to provide a split at about 48 mesh, such adjustment being effected by selection of the head or pressure maintained at the lower end of the hindered-settling column, as will be understood by persons familiar with hydraulic classification. Hence the principal effect of the hydraulic classifier, in sizing, was that the product continuously discharged represented material from predominantly +48 mesh, while the tailings, withdrawn from the upper column of the apparatus represented predominantly -48 mesh material, e.g. with not more than 20% to 25% +48 mesh.

The hydraulic sizer product was found to have a grade of about 75% B.P.L. and constituted high grade phosphate concentrate.

In these operations there was some overflow from the classifier, removing excess water and carrying some minor amount of fines or slimes, all of which was discarded to debris.

The classifier tailings, having a grade between 65% and 70% B.P.L., corresponding roughly to the grade of the classifier feed, were then given a second rinsing. Although it is conceivable that the hydraulic classifying may afford sufficient rinsing in itself, it is deemed preferable that for completeness of removal of the negative-ion reagents and the acid used for such removal or de-oiling, there be two rinsing steps. Hence this second rinsing operation, now needed only for the tailings fraction, was effected with appropriate addition and removal of water in suitable equipment, such as a Dorr rake classifier. This last operation is not such as to have any classifying function, although it accomplishes some de-watering as appropriate for the subsequent flotation.

The rinsed and de-watered pulp, being the tailings from the hydraulic classifier, is then treated with positive-ion reagent, and subjected to flotation in an appropriate cell, e.g. an Airflow machine. Although various positive-ion reagents may be employed, i.e. as well known in the art, for selectively floating silica or siliceous material from admixture with phosphate, it is preferred to use an amine reagent, and more particularly an amine reagent of the character described in U.S. Patent No. 2,553,905, granted May 22, 1951, to R. D. Evans. The preferred amine reagent, in accordance with the cited patent, comprises a soluble salt of an amine, such as an aliphatic amine of at least moderately long chain, which for purposes of use is treated with sodium hydroxide immediately prior to its introduction in the pulp, the amine-caustic mixture being augmented with kerosene, e.g. as described in the last-cited patent. It is understood that as so prepared the amine actually introduced in the pulp is in a finely divided, essentially insoluble form, yet so circumstanced as to be exceptionally effective for the desired silica-selecting operation.

The flotation cells were operated with the pulp so treated, yielding a froth flotation of the siliceous material, and a non-floated discharge which constituted high grade phosphate concentrate, specifically, the concentrate graded about 75% or better B.P.L., while the silica float or tailing contained only about 15% to 20% B.P.L. This last concentrate was, in practice, combined with the settling product of the hydraulic classifier, to afford a complete phosphate concentrate having a grade of about 75% and thus suitable for a variety of uses to which such material may be put, e.g. manufacture of phosphate fertilizers, or for making phosphoric acid or other phosphates as by various procedures. The silica tailings from the final flotation stage were discarded to the debris piles or ponds of the plant.

It will be seen that the described procedure, including the hydraulic classifying step where the classifying de-

vice functions as both sizer and rinser, and including the sand-flotation as applied only to the sizer tailing, afforded a highly satisfactory concentration of phosphate from a relatively low grade feed. These results were accomplished with economy of equipment and reagent, particularly in that the hydraulic classifier is considerably simpler and less costly than flotation cells of like capacity, and particularly since the amount of the expensive amine reagent required for the silica flotation is markedly reduced. In contrast, moreover, with a prior operation which dispensed with silica flotation and instead subjected the rougher concentrate to a second, negative-ion, cleaning flotation, with the cleaning cell tailings (as middlings) returned to the roughing cell feed, the metallurgical results of the new process represented appreciable improvement. In such prior operations, the final (cleaning cell) concentrate ranged around 70% B.P.L. or slightly more, whereas with the present process, the product grade was about 75% B.P.L.

As noted, the process involves recovery of a considerable amount of high grade phosphate concentrate directly as the hydraulic classifier product. The actual amount so recovered varies considerably with the particle size and other characteristics of the feed, but is usually at least about 10% of the rougher concentrate (both measured as solids by weight) and in some cases was as high as 40% to 50% of the rougher concentrate.

As a second example of the process, reference may be had to a fraction of phosphate ore mined in another Florida area, e.g. near Pierce, Florida. Such ore was treated first by a washing operation to recover high grade phosphate in +14 mesh sizes, the -14 mesh material being treated in a hydraulic classifier, and yielding a product, chiefly +65 mesh material, to which the present process was applied. The tailings from the above-mentioned primary sizer were extremely low grade, and of relatively fine particle size. The feed for the improved process in this instance thus represented -14 mesh mineral, including particle sizes down to about 65 mesh, i.e. considerably coarser than the material employed for feed in the first example, above. In this second example the feed material had a grade of about 40% B.P.L.

Such material was again mixed with negative-ion reagent material, viz. tall oil, fuel oil and caustic, and supplied continuously to a belt concentrator, e.g. a concentrating belt as described above, operated with appropriate supplementary water for promoting sidewise flow of the phosphate on the belts. The collected rougher concentrate had a grade of about 65% B.P.L., while the discarded tailing represented chiefly the larger size particles of silica. This rougher concentrate was then treated in exactly the same manner as the rougher concentrate in the first example, being subjected to sulfuric acid in an appropriate agitator, for de-oiling, and then being put through an Evans hydraulic sizer, from which the sizer product was withdrawn at the foot of the hindered-settling column and the sizer tailings at the head of the upper or free-settling column. This product consisted of good grade phosphate concentrate, i.e. about 70% B.P.L. or better. The sizer tailing was rinsed again, with appropriate de-watering, and was treated with the described amine reagent and subjected to flotation for separation of a silica float. The floated siliceous material contained only about 13% B.P.L. while the discharged concentrate from the flotation cells contained about 71% B.P.L. and was added to the product of the hydraulic sizer. In these operations the sizer product represented about 45% of the rougher concentrate from the rougher belts, while the product from the silica flotation machine represented a further 45% (approximately) of the rougher concentrate. As in the first example, the process thus represented an effective and economical operation, yielding a good grade of phosphate concentrate. Such operation thus dispensed with the so-called cleaning belts, which are a

relatively costly installation, as well as supplemental equipment utilized or required for such apparatus.

In specific tests to which the above examples are particularly related, the proportion of the rougher concentrate which was in effect separated as final product by the hydraulic classifier was from 10% to 20% in the case of the concentrate from flotation roughing and was about 45% for the concentrate from belt roughing. The difference in these specific instances arose because the belt rougher concentrate contained more coarse material than the flotation rougher concentrate, but it will be understood that these effects are a result of specific conditions and that with other particle size characteristics of the original feed and rougher concentrations, correspondingly different proportions of the sizer feed will appear as the sizer product of high grade phosphate.

Whereas in the examples, the hydraulic classifier was operated to fractionate the pulp roughly at the point of about 48 mesh, other points of split or sizing may be adopted as circumstances dictate. For example, it may be feasible in some cases to adjust the split for a somewhat smaller particle size, i.e. so that the sizer product would contain a substantial amount of particles at least a little finer than 48 mesh. On the other hand, the segregation may be adjusted to be primarily effected at a coarser particle size, e.g. even up to about 35 mesh or so. In general, the process is applicable where the rougher feed contains substantial quantities of material both coarser and finer than the selected point of sizer split, and thus where the rougher concentrate contains substantial quantities of phosphate particles coarser than such point and significant amounts of both phosphate and siliceous material in the finer sizes. Stated in a more particular sense, the procedure is especially appropriate where the sizer split can be made at a point in a range of particle sizes definable as from 35 to 55 mesh, or preferably 40 to 50 mesh, where at least about 10%, say, of the rougher feed is in each of the four categories of phosphate and siliceous particles larger and smaller than the selected point of sizer split, or where correspondingly at least about 10% to 15% or so of the sizer feed is in each of the three categories of coarse and fine phosphate and fine silica. It will be understood that in the foregoing and other references to amounts or percentages of feed, concentrate, tailings and the like, the stated values are given for, or based upon, the dry weight of the respective materials.

As will be understood, the nature and amounts of the various reagents employed for the roughing and silica separating concentrations can be readily selected in accordance with principles well known in the art. Thus as indicated above, negative-ion reagents, usually fatty acid materials, of phosphate-floating or phosphate-selecting character are well known, as likewise are the positive-ion reagents, most usually amines, that have silica-floating or silica-selecting properties in a pulp consisting of phosphate particles and siliceous gangue. By way of specific instance, in the first of the above examples, effective operation for a rougher flotation feed that had a grade of 24% B.P.L. and contained 53% larger than 48 mesh and 47% smaller, the following approximate amounts of negative-ion reagents were used per long ton of the feed (dry weight): tall oil 2.35 pounds, fuel oil 3.06 pounds, and caustic 0.44 pound. The rougher concentrate (floated) was about 25% by weight of the rougher feed, while the rougher tailings contained 64% larger than 48 mesh, chiefly silica, as indicated above.

In the same example, a specific instance of satisfactory feed of positive-ion reagents for the silica flotation operation on the sizer tailings involved the following, relatively modest amounts of reagents per long ton of such tailings: about 0.1 pound of water-soluble amine salt, with which was incorporated, immediately before use, about 0.04 pound of sodium hydroxide (supplied in solution)

and about 0.25 pound of kerosene. The selected amine reagent was a composition understood to consist essentially of a mixture of octadecyl and similar amine acetates, prepared by the neutralization of primary tallow amine with acetic acid, and being a General Mills product known as Alamac 26. In all examples herein, the de-oiling reagent was sulfuric acid (76%), selected as being most conveniently available, and used in the proportion of about 4 pounds per long ton of rougher concentrate.

In the example of the process where belt concentration was employed for roughing, the negative-ion reagents, per long ton, were, in approximate amounts: tall oil 1.0 pound, fuel oil 1.5 pounds, and caustic 0.3 pound. Again, in such example, the silica flotation was effected with the same combination of positive-ion reagent material, prepared with the following amounts per long ton of sizer tailings: Alamac 26 (amine) 0.336 pound, sodium hydroxide 0.067 pound, and kerosene 0.53 pound. These last amounts were equivalent to only 0.187 pound of Alamac 26, 0.037 pound of sodium hydroxide and 0.30 pound of kerosene, per long ton of the rougher concentrate.

As further indication of the effective action of the hydraulic classifier, i.e. the sizer-rinser in the process of the invention, the following table gives a representative set of results obtained in one test of the first example, analyzed by particle size (references to mesh being of plus significance unless otherwise noted):

Table I
SIZER-RINSER FEED

Mesh	Percent Wt.	Cum., Percent Wt.	Percent BPL	Cum., Percent BPL
20-----	Trace	1.0	72.79	72.79
28-----	1.0	6.6	75.85	75.39
35-----	5.6	27.1	74.83	74.98
48-----	20.5	59.4	70.63	72.61
65-----	32.3	90.2	63.82	69.61
100-----	30.8	100.0	46.00	67.30
-100-----	9.8			

SIZER-RINSER PRODUCT

20-----	1.2	1.2	66.20	66.20
28-----	10.5	11.7	73.35	72.62
35-----	43.9	55.6	75.28	74.72
48-----	36.1	91.7	76.08	75.26
65-----	6.5	98.2	73.47	75.14
100-----	1.5	99.7	68.13	75.01
-100-----	0.3	100.0		

SIZER-RINSER TAILING

20-----	Trace	0.6	73.35	73.35
28-----	0.6	3.8	74.03	73.90
35-----	3.2	20.0	69.95	71.49
48-----	16.2	51.3	64.95	68.72
65-----	31.3	89.0	54.16	67.12
100-----	37.7	100.0		
-100-----	11.0			

As indicated above, the subsequent silica flotation separated the fine siliceous material, yielding a non-floated concentrate with a grade of about 75% B.P.L. This was added to the sizer-rinser product, of like grade. The total phosphate recovery was about 70% of the phosphate (B.P.L.) in the original rougher feed, while the total amount of the combined product represented about 80% of the weight of the sizer-rinser feed, i.e. the rougher concentrate. About 90% of the B.P.L. units in the rougher concentrate were recovered in the combined high grade product.

In certain specific tests of the second example, where the roughing operation was a belt concentration, and where grades and amounts of certain feeds or other fractions were as stated above in the example, the ultimate combined concentrate represented 90.5% by weight of the rougher concentrate (sizer feed) and contained 97.8% of the phosphate (B.P.L.) originally present in

such rougher concentrate. The sizer tailing had a grade of about 61.6% B.P.L., while the final combined product graded about 71% B.P.L.

References in the claims to concentrate fractions or products as having a greater phosphate content than the rougher concentrate, means that such fractions or products are of higher grade, i.e. have a higher percentage of phosphate per unit weight of solids.

It is to be understood that the invention is not limited to the specific examples hereinabove described, but may be carried out in other ways without departure from its spirit.

I claim:

1. A process of concentrating phosphate-containing mineral material which comprises both coarse and fine phosphate and siliceous particles, comprising subjecting said material to roughing concentration in water with negative-ion reagent material to yield a phosphate-enriched concentrate having a reduced content of coarse siliceous particles, subjecting said concentrate, in aqueous pulp, to hydraulic classification by settling while adding water, for separating a coarse settled product having a greater phosphate content than said concentrate and for producing a rinsed phosphate-containing tailing including fine siliceous particles, said hydraulic classification comprising effecting downward settling of coarse particles from the solids of said pulp, and collection of the aforesaid coarse settled product at a lowermost locality, while directing said additional water continuously upward through the region of said downward settling and into a locality thereabove while segregating and carrying away the said rinsed tailing of fine particles from said last-mentioned locality and separately removing excess water as overflow, and subjecting said tailing to silica-removing concentration in water with positive-ion reagent material to separate siliceous particles and produce a concentrate having a greater phosphate content than the first-mentioned concentrate.

2. In a process of concentrating phosphate-containing mineral material consisting essentially of both coarse and fine phosphate and impurity particles, including fine siliceous particles, the steps of subjecting said material to roughing concentration in water with negative-ion reagent material to separate a tailing comprising coarse impurity particles and to yield a phosphate-enriched concentrate, subjecting said concentrate, in aqueous pulp, to hydraulic classification with added water, to separate a settled product comprising coarse phosphate particles and to provide a rinsed tailing comprising fine phosphate and fine siliceous particles, said hydraulic classification comprising effecting downward settling of coarse particles from the solids of said pulp, and collection of the aforesaid coarse settled product at a lowermost locality, while directing said additional water continuously upward through the region of said downward settling and into a locality thereabove and while segregating and carrying away the said rinsed tailing of fine particles from said last-mentioned locality and separately removing excess water as overflow, and subjecting said last-mentioned tailing to silica-removing concentration in water with positive-ion reagent material, to elevate and remove fine siliceous particles and to recover a concentrate product comprising fine phosphate particles.

3. In a process of concentrating phosphate-containing mineral material having a particle size less than about 14 mesh and consisting essentially of both coarse and fine phosphate particles and impurity particles, including fine siliceous particles, the steps of subjecting said material to roughing concentration in water with negative-ion reagent material to separate a tailing comprising coarse impurity particles and to yield a phosphate-enriched concentrate, treating said concentrate with acid for removal of said reagent material, subjecting said concentrate, in aqueous pulp, to hydraulic classification by settling while adding water, for separating a settled product comprising

coarse phosphate particles and having a greater phosphate content than said concentrate and for producing a rinsed tailing comprising fine phosphate and fine siliceous particles, said hydraulic classification comprising effecting downward settling of coarse particles from the solids of said pulp, and collection of the aforesaid coarse settled product at a lowermost locality, while directing said additional water continuously upward through the region of said downward settling and into a locality thereabove and while segregating and carrying away the said rinsed tailing of fine particles from said last-mentioned locality and separately removing excess water as overflow, and subjecting said last-mentioned tailing to silica-removing concentration in water with silica-floating positive-ion reagent material, to elevate and remove fine siliceous particles and to produce a concentrate product having a greater phosphate content than said first-mentioned phosphate-enriched concentrate.

4. In a process of concentrating phosphate-containing mineral material which has a particle size less than about 14 mesh and which comprises both coarse and fine phosphate particles and both coarse and fine siliceous particles, the steps of subjecting said mineral material, as an aqueous pulp, to a roughing concentration with negative-ion reagent material to yield a phosphate-enriched concentrate comprising coarse and fine phosphate particles and fine siliceous particles, and a tailing comprising coarse siliceous particles, treating the concentrate with de-oiling reagent to remove said first-mentioned reagent material, subjecting said treated concentrate in aqueous pulp form to hydraulic classification including effectuating settling while supplying additional water to the concentrate pulp, to separate a settled product comprising coarse phosphate particles and to produce a rinsed tailing comprising fine phosphate and siliceous particles, said hydraulic classification comprising effecting downward settling of coarse particles from the solids of said pulp, and collection of the aforesaid coarse settled product at a lowermost locality, while directing said additional water continuously upward through the region of said downward settling and into a locality thereabove and while segregating and carrying away the said rinsed tailing of fine particles from said last-mentioned locality and separately removing excess water as overflow, and subjecting said last-mentioned tailing to floatation with positive-ion reagent material to separate a float of fine siliceous particles and recover a concentrate of fine phosphate particles, said settled product and said last-mentioned concentrate comprising phosphate-enriched product material having a substantially higher phosphate content than the aforesaid mineral.

5. In a process of concentrating phosphate-containing mineral material which has a particle size less than about 14 mesh and which comprises both coarse and fine phosphate particles and both coarse and fine siliceous particles, the steps of subjecting said mineral material, as an aqueous pulp, to a roughing concentration with negative-ion reagent material to yield a phosphate-enriched concentrate comprising coarse and fine phosphate particles and fine siliceous particles, and a tailing comprising coarse

siliceous particles, treating the concentrate with acid to remove the aforesaid negative-ion reagent material, subjecting said concentrate, in aqueous pulp, to hydraulic classification by settling while adding water, for separating a settled product comprising coarse phosphate particles and having a greater phosphate content than said concentrate and for producing a rinsed tailing comprising fine phosphate and fine siliceous particles, said hydraulic classification comprising effecting downward settling of coarse particles from the solids of said pulp, and collection of the aforesaid coarse settled product at a lowermost locality, while directing said additional water continuously upward through the region of said downward settling and into a locality thereabove and while segregating and carrying away the said rinsed tailing of fine particles from said last-mentioned locality and separately removing excess water as overflow, and subjecting said last-mentioned tailing to silica-removing concentration in water with silica-floating positive-ion reagent material, to elevate and remove fine siliceous particles and to produce a non-floated phosphate concentrate having a greater phosphate content than said first-mentioned concentrate.

6. A process as defined in claim 5, wherein the silica-removing concentration comprises a froth-flotation operation employing silica-selecting amine reagent material for separating a silica float.

7. A process as defined in claim 6, wherein the first-mentioned roughing concentration comprises a flotation operation employing phosphate-selecting fatty acid reagent material for elevating said first-mentioned phosphate-enriched concentrate.

8. A process as defined in claim 5, wherein the first-mentioned roughing concentration comprises belt concentration employing phosphate-selecting fatty acid reagent material for promoting separation from the belt of said first-mentioned phosphate-enriched concentrate, and wherein the silica-removing concentration comprises a froth-flotation operation employing silica-selecting amine reagent material for separating a silica float.

9. A process as defined in claim 5, wherein the phosphate-containing mineral material contains particles throughout a range from +28 mesh to -65 mesh, the coarse particles being +48 mesh and the fine particles being -48 mesh, and wherein said hydraulic classification is effective for sizing division to provide the aforesaid settled product having particle size predominately +48 mesh and the aforesaid second-mentioned tailing having particle size predominately -48 mesh.

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