Dec. 10, 1957 C. A. HOLLINGSWORTH 2,815,859

CONCENTRATION OF PHOSPHATIC MATERIAL

Filed Oct. 15, 1954

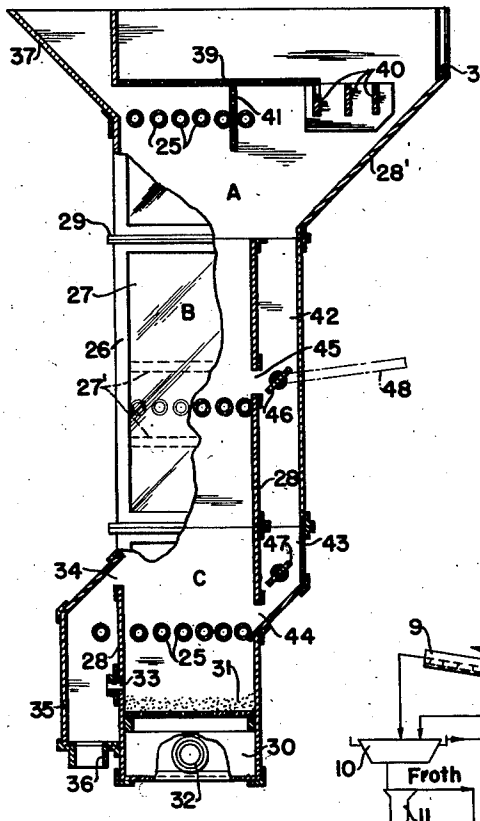



FIG. 2

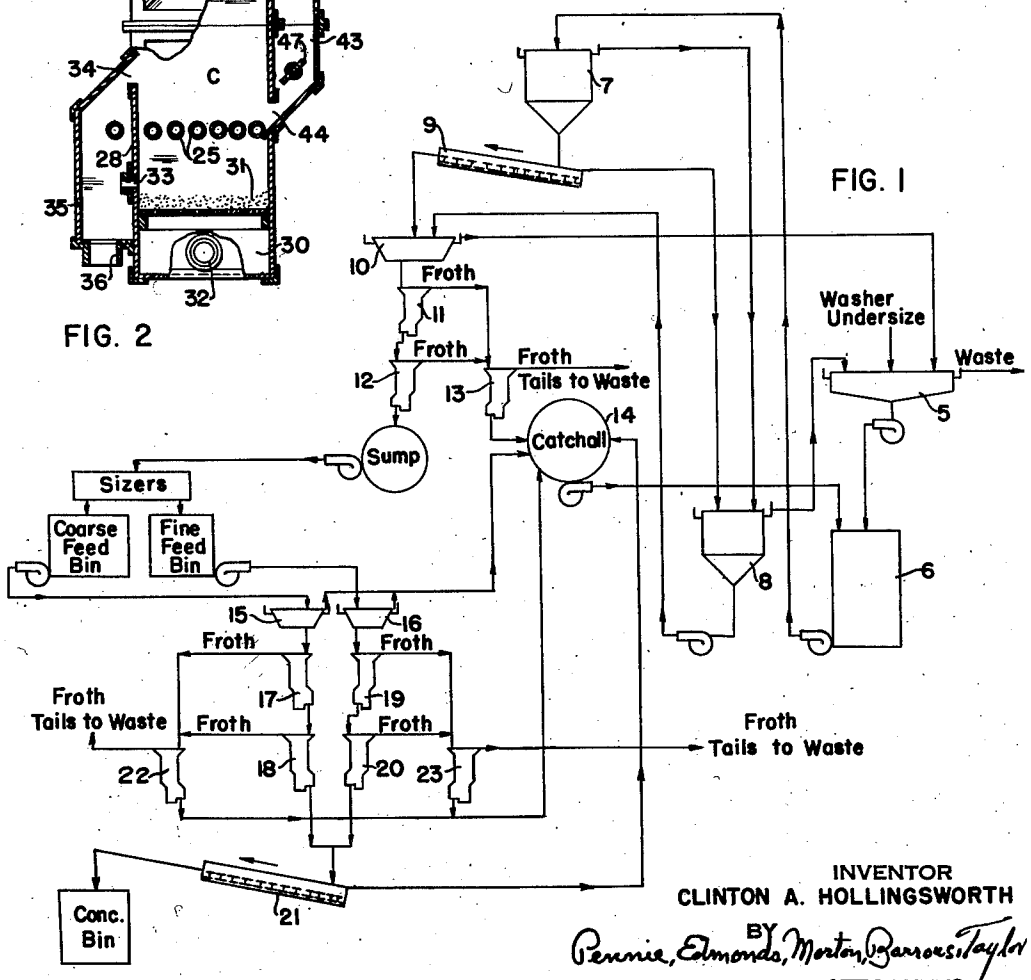



FIG. 1

INVENTOR
CLINTON A. HOLLINGSWORTH
BY
Pennie, Edmonds, Morton, Barrows & Taylor
ATTORNEYS

United States Patent Office

2,815,859
Patented Dec. 10, 1957



2,815,859

# CONCENTRATION OF PHOSPHATIC MATERIAL

Clinton A. Hollingsworth, Lakeland, Fla., assignor to Smith-Douglass Company, Incorporated, Norfolk, Va., a corporation of Virginia

Application October 15, 1954, Serial No. 462,470

8 Claims. (Cl. 209—167)

This invention relates to the froth flotation process for concentrating phosphatic material and more particularly to the separation of silica from phosphatic material by froth flotation, and has for its object the provision of an improved method of froth flotation for separating silica from phosphatic material.

The main phosphatic constituent of Florida phosphate rock, as well as most native phosphate minerals containing fluorine, is fluorapatite (usually referred to as apatite) commonly believed to be a combination of tricalcium phosphate and calcium fluoride ($Ca_{10}F_2(PO_4)_6$). The grade of the rock is determined by its tricalcium phosphate content, which is usually designated in the industry as bone phosphate of lime (BPL). Silica is one of the chief valueless or gangue constituents of phosphate rock and in the phosphate industry is commonly determined and reported as insolubles (insol.). For many purposes, such as the defluorination of phosphate rock by calcination without fusion, a phosphatic raw material of good grade and low silica content, say less than 4% and preferably not exceeding 3% insol., is desirable.

In the washing and concentration of phosphate rock, it is presently the common practice throughout the Florida phosphate field to double-float the undersize from the washing plant. That is, after concentration of the raw rock by washing and the like, the undersize or fines from the washer is further concentrated in two stages of froth flotation. In the first stage, the deslimed flotation feed is conditioned with caustic soda, fuel oil and a fatty acid such as tall oil, and the conditioned feed is subjected to froth flotation where a phosphate product (commonly called a rougher concentrate) is floated and the underflow (largely silica) is discarded to waste. The rougher concentrate normally contains 8 to 30% of silica, and hence its phosphate grade (BPL) is too low to be of much practical value. Accordingly, the rougher concentrate is de-oiled by scrubbing with sulphuric acid followed by desliming. The de-oiled product (commonly called the "amine cell feed") is subjected to the second stage of froth flotation in the presence of a strong cationic collecting agent, such as an amine, where silica is floated and discarded to waste. The underflow of the second stage of flotation is a high grade phosphate product of low silica content, and is transferred to storage. In the phosphate field, the phosphate product is called the concentrate regardless of whether it is the floated product or the underflow, and hence the silica float is called "tailings" or "tails."

Phosphate ores are friable and tend to produce deleterious amounts of slime during flotation treatment, especially in machines of the agitation type. Slime has been found to be particularly objectionable in the amine circuit flotation, since the presence of slime necessitates an increased amount of the relatively costly amine reagent with an attendant loss of phosphatic material in the silica tailings in order to obtain the desired low silica content in the phosphate concentrate. In the aforementioned two-stage practice, considerable slimes are produced in the



scrubbing of the rougher phosphate concentrate with sulphuric acid, and hence the de-oiled product is deslimed preparatory to the second stage of froth flotation. But even then, the types of flotation apparatus presently used in the Florida phosphate field produce objectionable amounts of slime during the flotation treatment due to the violent agitating action of the impeller.

In my copending application for Letters Patent of the United States Ser. No. 452,177, filed August 25, 1954 (Patent No. 2,758,714), I have described a method of pneumatic-hydraulic flotation peculiarly adapted for phosphate minerals because of its highly effective aeration without creating slime in objectionable amount. I have found that when flotation is carried out in accordance with this method, the first stage of the present common flotation practice (in which phosphate is floated) can be replaced by a rougher froth flotation step in which silica is floated, thereby eliminating the present de-oiling treatment of the rougher phosphate concentrate. The present invention makes use of this discovery, and in its broad aspect involves an improved method of separating silica, as a float, from phosphatic material by froth flotation in the presence of one or more appropriate cationic collecting flotation agents. In accordance with the method of the invention, the underflow of the first step or stage of froth flotation is a rougher phosphate concentrate, which is further treated or cleaned in the second step of froth flotation similar in character to the first step. Both steps of froth flotation are carried out in the presence of a strong cationic collecting agent and silica is floated in each. Each step of flotation is carried out in a relatively deep column of pulp and the downward velocity of pulp flow in the column is substantially decreased and the creation of slimes is thereby minimized by introducing water in a fine state of subdivision into the pulp column through a porous medium at the bottom thereof, in accordance with the invention of my aforementioned application. In addition to providing two separate and similar steps of froth flotation, in which different cationic flotation agents may be used if desired, the method of the invention contemplates particle sizing of the rougher phosphate concentrate into a coarse feed and a fine feed for the second step of froth flotation, thereby permitting, if desired or necessary for optimum results, the use of different cationic flotation agents with the feeds of different particle size.

The foregoing and other novel features of the invention will be better understood from the following description taken in conjunction with the accompanying drawings, in which:

Fig. 1 is a diagrammatic flow sheet embodying the principles of the invention, and

Fig. 2 is a side elevation, mostly in section, of the pneumatic-hydraulic flotation cell or apparatus described in my aforementioned application and especially adapted for practicing the present invention.

Referring to the flowsheet of Fig. 1, the feed to the flotation plant is the undersize or fines from the phosphate washer plant having a particle size normally minus 14 mesh, i. e. substantially all through a 14 mesh standard Tyler screen. The following is a typical screen analysis of the flotation plant feed or heads:

| +20 mesh | −20 +35 | −35 +65 | −65 +150 | −150 |
|---|---|---|---|---|
| 7.2% | 18.0 | 50.8 | 21.9 | 2.1 |

Thus, the flotation heads or feed is predominately (i. e. more than half) minus 20 mesh and on 65 mesh. The BPL of this feed will vary from 20 to 50%, and usually the silica content (insol.) exceeds the phosphate content

(BPL). The washer undersize is first dewatered in a large hydroseparator **5**, the overflow of which is discharged to waste, and the underflow pulp is pumped to storage bins **6**. From the storage bins, the pulp is pumped, as required, to a surge bin **7**, the overflow of which goes to a relatively small hydroseparator **8**. The underflow of the surge bin goes to a rake classifier **9**, the overflow of which goes to the hydroseparator **8**. The rake product is delivered to a deslimer **10**, the overflow of which is returned to the large hydroseparator **5**. The settled underflow of the hydroseparator **8** is pumped to the deslimer **10**.

The deslimer pulp is delivered to two series (**11** and **12**) of rougher flotation cells, arranged in tandem, that is the phosphate underflow of the cells **11** is the feed of the cells **12**. The silica froths of both cells **11** and **12** are combined and fed to froth-cleaning flotation cells **13**. The silica froth (tails) of the cells **13** is discharged to waste, and the underflow of these cells is delivered to a catchall sump or middlings tank **14**.

The underflow of the cells **12** (rougher phosphate concentrate) is delivered to a storage sump, from which it is pumped, as required, to particle sizers in which products of two different particle sizes are produced, namely coarse and fine, as feed for the flotation cleaner cells. The following are typical screen analysis of these sized products:

| | +20 mesh | −20 +35 | −35 +65 | −65 +150 | −150 |
|---|---|---|---|---|---|
| Coarse feed, percent | 21.6 | 54.3 | 17.9 | 6.2 | ------ |
| Fine feed, percent | ------ | 1.5 | 24.8 | 68.5 | 5.2 |

Thus, the coarse feed is predominately (i. e. more than half) minus 14 mesh and on 35 mesh, while the fine feed is substantially all minus 35 mesh and predominately minus 65 mesh.

The coarse and fine feeds are delivered to separate rinsers **15** and **16**, respectively, in the nature of deslimers. The underflow of the coarse feed rinser **15** is delivered to two series (**17** and **18**) of flotation cells (cleaners) arranged in tandem. The underflow of the fine feed rinser **16** is delivered to two series (**19** and **20**) of flotation cells (cleaners) arranged in tandem. The underflows of the cells **18** and **20** (final cleaner concentrate) are combined and deliverd to a dewatering screw classifier **21**, and the dewatered phosphate concentrate is discharged to storage bins. The screw classifier overflow is delivered to the middlings tank **14**.

The silica froths of the cells **17** and **18** are combined and retreated in a froth cleaning cell **22**, while the silica froths of the cells **19** and **20** are combined and retreated in a froth cleaning cell **23**. The silica froths (tails) of the cells **22** and **23** are discharged to waste, and the underflow of these cells is conveyed to the middlings tank **14**. Middlings from the tank **14** are pumped to the storage bins **6** for the dewatered washer undersize at the head of the flotation plant.

As a general indication of the distribution of the flotation cells in the plant, where there are 6 rougher cells in each series **11** and **12**, 4 froth-cleaning cells **13** (of approximately the same size) are provided. In each of the series of cleaning cells (**17—18** and **19—20**) there are 2 cells, and 1 froth cleaning cell (**22** and **23**) is provided for each series of cleaning cells.

The pneumatic hydraulic flotation apparatus or cell illustrated in Fig. 2 of the drawings is built in separate units or sections which are vertically assembled and secured together to form the complete apparatus. The apparatus illustrated in the drawings is built up of a top unit A, an intermediate unit B and a bottom unit C. The top and bottom units may advantageously be about 2 feet in depth and the depth of the intermediate unit (or units) may then advantageously be from 2 to 3 feet. As many intermediate units B may be assembled in the complete apparatus as required to give the desired over-all depth of pulp column. Usually the assembled cell will accommodate a pulp column of at least six feet in depth. Each unit is provided with at least one group of horizontally disposed air-diffusers **25**, so that when the units are assembled the various groups of air-diffusers are superposed and vertically spaced. The air-diffusers are of such construction that air is uniformly introduced into the pulp column in a very finely disseminated state over the entire cross-sectional area of the pulp column.

Each unit comprises a metal frame **26** to which are bolted or otherwise appropriately secured side and end members **27** and **28**, respectively. The side and end members may advantageously be transparent thermoplastic resin plates, such as Rohm and Haas Company's "Plexiglas," through which the action of the air-diffusers and the condition of the pulp column can readily be observed. Where the side and end members are of metal or other opaque material, the side members are preferably provided with transparent windows at levels opposite the air-diffusers, as indicated by the dotted lines **27'**. The juxtaposed ends of the frames **26** of adjacent units have peripheral flanges **29** for bolting or otherwise appropriately securing the units together.

The bottom unit C has a hydraulic compartment **30** at its lower end which communicates with the main upper portion of the unit through a porous medium **31**. While the porous medium is shown in the drawings as consisting of a punched metal plate covered with a bed of lead shot about one inch in depth, the medium may consist of porous tile, punched metal plate, metal screen, heavy canvas and the like. The hydraulic compartment **30** is connected to a suitable source of water through a pipe **32** having a suitable control valve (not shown).

One end wall **28** of the bottom unit C has a lower pulp discharge opening **33** and an upper pulp discharge opening **34** communicating with a common pulp discharge chute **35**. The discharge opening **33** is just above the porous medium **31**, while the discharge opening **34** is near the top of the unit, and the horizontally disposed air-diffusers are about midway between the two openings with one diffuser positioned in the discharge chute **35**. The chute **35** extends across the width of the end wall **28**, and is provided at its bottom with a pulp discharge pipe **36**. The rate at which water is introduced into the hydraulic compartment **30** is so proportioned with respect to the openings **33** and **34** and the pipe **36** as to be substantially the same as the rate at which water is discharged in the mineral pulp through the pipe **36**.

The top unit A has a feed hopper **37** at the top of one end wall and an adjustable froth overflow weir **38** at the top of the outwardly flaring opposite end wall **28'**. A perforated plate **39** is horizontally positioned a short distance (normally about 2 inches) below the level of the weir **38**, and extends from the inside bottom of the feed hopper **37** to the first of three vertically disposed (i. e. depending) and horizontally spaced transverse baffles **40**. The outward flare of the end wall **28'** is such that the top of the unit A is about twice the length of the bottom of the unit, and the horizontally spaced baffles **40** are positioned above the flared end wall. A transverse perforated baffle **41** depends from about the center of the horizontal perforated plate **39**, with one air-diffuser (nearest the discharge weir **38**) on one side of the baffle **41** and the other air-diffusers of unit A on the outer, or feed hopper, side of the baffle **41**.

The intermediate unit B is provided with a vertical chamber or channel **42** along that end wall adjacent the flared end wall **28'** of the top unit. The channel **42** communicates at its top with the top unit A and at its bottom with a similar but shorter channel **43** at the upper end of the bottom unit C. The channel **43** communicates through an opening **44** with the unit C at a level slightly above that of the group of air-diffusers of the unit, and the channel **42** communicates through an opening **45** with the

unit B at a level slightly above that of the group of air-diffusers in the unit. The channels 42 and 43 are of the same width as their respective units, and are provided with transverse dampers 46 and 47, respectively, for regulating the pulp circulation. The dampers are adapted to be adjusted by a removable wrench 48, shown in dotted lines.

Each of the flotation cells in the flow sheet of Fig. 1 is of the type shown in Fig. 2. In each the feed of phosphatic material, in the form of an aqueous mineral pulp, is delivered through the hopper 37 into a relatively deep column or body of pulp undergoing froth flotation treatment. The flotation reagent (or reagents) may be mixed with the pulp before or as it is introduced into the hopper, or part or all of the reagent may be introduced at one or more lower levels in the pulp column. Where the pulp has been well deslimed, it is preferable to add the reagent with the feed, but when the feed contains any slime, some or all of the reagent should be introduced at one or more lower levels in the pulp column. Aeration takes place at the three (or more) different levels at which the groups of air-diffusers are positioned, and is extensive in sectional area and gentle and uniform in character. Each group of air-diffusers discharges a dense mass of extremely fine, uniform and gentle streams of air into the pulp column over the entire cross-sectional area thereof.

Bubbles from the rising air streams attach themselves to the silica particles selectively conditioned by the cationic flotation agent to float, and carry such particles into a layer or column of froth overlying the surface of the pulp body. The perforated plate 39 is at approximately the liquid level of the pulp column, and serves to minimize surface turbulence so that the overlying layer of froth is relatively quiescent. As the froth passes longitudinally beyond the active influence of the top air-diffusers toward the overflow weir 38, it moves over a relative quiescent body of pulp (adjacent and in direct communication with the pulp column) where opportunity is afforded phosphate particles to drop out of the froth layer. Thus in effect there are in the top unit A an active aerated zone and an adjacent quiescent settling zone. The single air-diffuser in the latter zone provides aeration for returning to the froth layer any silica particles in the immediate vicinity of the boundary between the two zones. The depending transverse baffles 40 and 41 divert descending phosphate particles away from the active aerated zone of the pulp column and generally in the direction of the open top of the channel 42. The baffle 41 extends more deeply into the pulp column than do the baffles 40, because of its proximity to the air-diffusers. The depending baffles 40 and 41 further tend to minimize circulation of the pulp body directly below the froth layer, and thus induce a quiescence in the pulp body that is beneficial in guiding the phosphate particles (dropped from the froth layer) toward the open end of the channel 42.

There is a natural downward circulation of pulp in the channels 42 and 43, due to the upward impetus that is given the pulp column in the units B and C by the rising air streams from the diffusers 25 in those units. By adjustment of the dampers 46 and 47, any desired portion of the downwardly circulating pulp can be diverted from the channels 42 and 43 through the openings 45 and 44 into the units B and C, respectively. Thus, the channels provide means for short-circuiting phosphate particles to a lower level of the pulp column, and for returning any silica particles to the pulp column, at levels of active aeration. In this manner, as well as in the characteristic arrangement of the air diffusers at several different levels in a relatively deep pulp column, silica particles are given several opportunities of attaching themselves to rising air bubbles thereby minimizing the amount of such particles included in the underflow.

The introduction of a controlled amount of water from the hydraulic compartment 30 into the lower portion of the pulp column is a characteristic feature of the method. The water so introduced into the pulp column is conveniently called the hydraulic water. It promotes circulation within the pulp column and prolongs the period of suspension of silica particles and gives them better, longer and more frequent opportunity for attachment to rising air bubbles. The hydraulic water also prevents sanding-up of the cell, since the pulp density of the underflow discharge is lowered by the water entering the pulp column in the region of the underflow discharge. The pulp density of the feed to the cells 11, 17 and 19 is generally within the range of 30 to 75% solids and is higher than the pulp density of their underflow discharge, which is generally within the range of 20 to 40% solids. Generally speaking, the average pulp density within the cells is roughly between 15 and 35% solids. From 25 to 50% of the water of the pulp column in these cells is introduced with the feed, and the remaining percent (that is from 75 to 50%) is introduced as hydraulic water. The volume of hydraulic water introduced into the unit C (per unit of time) is substantially equivalent to the volume of water in the underflow discharge from the pipe 36 in the same time period.

As phosphate particles descend into the unit C, some will be carried by the pulp stream through the upper opening 34 into the discharge chute 35. The simple air-diffuser near the top of the chute offers a further and last opportunity for any silica particles to be carried upward by attachment to rising air bubbles. Similarly, the remainder of the lowermost air-diffusers in the unit C promote final cleaning of the pulp stream of silica particles before discharge of the pulp through the bottom opening 33 into the chute 35.

Withdrawing with the underflow discharge of substantially the same volume of water as introduced into the unit C through its porous bottom decreases the downward velocity of pulp flow and thus provides and insures a relatively quiescent column of pulp above the unit C. This relatively quiescent column of pulp provides better and longer contact of silica particles with air particles, and thus promotes separation of silica and phosphate particles and minimizes slime production. In this quiescent column of pulp there are only gentle circulatory motions resulting from the effects of the rising air streams and the channels 42 and 43, and hence little, if any, tendency to create slime.

Normally, the froth column or bed will be from 1 to 5 inches in depth. The depth of froth depends to a large extent upon the type of flotation reagents employed, some yielding a voluminous froth while others give very little froth. The depth of froth can be controlled to some extent by the use of different frothing agents, surface-active agents, depressants etc. The perforated froth-subduing plate 39 is normally about 2 inches below the level of the adjustable weir 38. The pulp (that is liquid) level may be raised or lowered, in relation to the perforated plate 29, by adjusting the number of filler-bars that make up the adjustable weir 28. Thus, the pulp level should be lowered for a shallow froth bed and raised for a deep froth bed.

It is presently the common practice in the Florida phosphate field to size the raw flotation feed (washer fines) in order to provide (in the second stage of flotation) a sized amine cell feed, since larger particles of silica do not float with the average quantity of reagents used, and an increase in the quantity of reagent required to float such larger silica particles results in floating large amounts of phosphate which are lost in the silica tails. In accordance with the present invention, the feed to the first bank of rougher flotation cells 11 is unsized washer fines, and a large proportion of the silica gangue is removed in this first step of flotation and before the rougher phosphate concentrate is sized preparatory to the second step of flotation. By dividing the rougher phosphate concentrate into products of two different sizes, different amounts of reagent, or different reagents, can be used with the

coarse feed and the fine feed to give the optimum result with each.

I have found that all silica does not appear to respond in the same way to flotation treatment. For example, one type of reagent floats part of the silica best while another type of reagent floats the remaining part of the silica best. By floating in two steps of silica flotation, advantage can be taken of this phenomenon. Thus, a different type of cationic collector may be used in the second or cleaner step of the invention than used in the first or rougher step, or perhaps different amounts of the same cationic collector in the two steps of flotation will give the optimum results. In the present preferred practice of the invention, the first step of flotation of unsized feed (in cells **11, 12** and **13**) is carried out with the reaction product of sulphuric acid and a mixture of a fatty amine and liquid hydrocarbon, such as described and claimed in the copending patent application of Jordan L. Wester and myself, Ser. No. 443,692, filed July 15, 1954. Typical of such a modified amine reagent is the reaction product of 4.76% (by weight) of sulphuric acid and a mixture of 19.05% of Armour and Co.'s "Armoflote S" (consisting of about 70% free fatty amine and about 30% nitrile) and 76.19% of crude turpentine. The second step of flotation of sized feed is then carried out with an unmodified amine (i. e. one not reacted upon by sulphuric acid), such as "Armoflote S" or other suitable fatty amine flotation reagent or other type of cationic collecting flotation agent, usually in conjunction with a suitable frothing agent such as pine oil, crude turpentine or one of the so-called alcohol frothers (e. g. di-isobutyl carbinol). Different quantities of the same unmodified amine, or different cationic collectors, may be used with the sized coarse feed and fine feed, respectively, to give the optimum result with each. While I now prefer cationic collectors of the amine type, other types of cationic collectors are now commercially available for floating silica and may be used in practicing the invention.

The following table illustrates the broad ranges of BPL and insol. in the principal products of the method, and also gives the more typical BPL and insol. contents generally obtained in plant practice.

| Product | Approx. BPL | Approx. Insol. |
|---|---|---|
| | *Percent* | *Percent* |
| Initial flotation feed | 20–50 | 73–38 |
| Generally (around) | 35 | 55 |
| Rougher phosphate conc. | 55–71 | 30–8 |
| Generally (around) | 65 | 15 |
| Silica float of cells 13 | 2–10 | 96–87 |
| Generally (around) | 6 | 92 |
| Coarse feed to cells 17 | 55–71 | 30–8 |
| Generally (around) | 65 | 15 |
| Fine feed to cells 19 | 55–71 | 30–8 |
| Generally (around) | 65 | 15 |
| Coarse phosphate conc. (cell 18) | 74–78 | 6–3 |
| Generally (around) | 76 | 4 |
| Fine phosphate conc. (cell 20) | 74–80 | 6–2 |
| Generally (around) | 77 | 3 |
| Coarse tails from cell 22 | 5–20 | 93–73 |
| Generally (around) | 10 | 88 |
| Fine tails from cell 25 | 5–20 | 93–73 |
| Generally (around) | 10 | 88 |

In the first rougher step of the invention, the BPL recovery is in excess of 90%, and usually 95% and higher. In the second cleaner step, the BPL recovery is usually at least 95% and frequently as high as 98–99%. The overall BPL recovery of the complete method, that is the amount of phosphate in the initial unsized washer undersize that is recovered in the final cleaner concentrate, is usually at least 85%, and frequently 90 to 95%. The slime loss in the method of the invention is for all practical purposes negligible, contrasted with a slime loss of about 10% in the heretofore common double float practice of the Florida phosphate field. The lost slimes have a relatively high BPL content, and consequently the overall BPL recovery of the flotation plant in the double float practice seldom exceeds 80%. The slimes are delivered to the pond, along with the silica tails, and no attempt is made to recover the phosphate in the bed of settled solids.

I claim:

1. The method of separating silica from an unsized silica-containing phosphate product which comprises subjecting the deslimed and unsized phosphate product to a first rougher step of pneumatic froth flotation in the presence of a cationic collecting flotation agent and in the course of which a silica froth and a rougher phosphate concentrate are obtained, subjecting said rougher phosphate concentrate to a sizing treatment to obtain a product of relatively coarse particle size and a product of relatively fine particle size, and subjecting each of said sized products to separate cleaner steps of pneumatic froth flotation in the presence of a cationic collecting flotation agent, each cleaner step of pneumatic froth flotation being conducted in the presence of a different cationic collecting flotation agent than that present in the preceding rougher step of pneumatic froth flotation.

2. The method of claim 1 in which the rougher step of pneumatic froth flotation is conducted in the presence of the reaction product of sulphuric acid and a mixture of a fatty amine with a liquid hydrocarbon and the cleaner step of pneumatic froth flotation is conducted in the presence of a fatty amine flotation agent unreacted upon by sulphuric acid.

3. The method according to claim 1 in which one of said sized products is subjected to a cleaner step of pneumatic froth flotation in the presence of the same cationic collecting flotation agent as that present in the preceding rougher step of pneumatic froth flotation, and in which the other of said sized products is subjected to a cleaner step of pneumatic froth flotation conducted in the presence of a different cationic collecting flotation agent than that present in the preceding rougher step of pneumatic froth flotation.

4. The method of separating silica from an unsized phosphate product containing at least two types of silica which comprises subjecting the deslimed and unsized phosphate product to a first rougher step of pneumatic froth flotation in the presence of a cationic collecting flotation agent and in the course of which a silica froth and a rougher phosphate concentrate are obtained, subjecting said rougher concentrate to a sizing treatment to obtain a product of relatively coarse particle size and a product of relatively fine particle size, and subjecting each of said sized products to separate cleaner steps of pneumatic froth flotation in the presence of a cationic collecting agent formulated to collect the type of silica predominating in each of said sized products.

5. The method of separating silica from an unsized phosphate product containing at least two types of silica which comprises subjecting the deslimed and unsized phosphate product to a first rougher step of pneumatic froth flotation in the presence of a cationic collecting flotation agent and in the course of which a silica froth and a rougher phosphate concentrate are obtained, subjecting said rougher concentrate to a sizing treatment to obtain a product of relatively coarse particle size predominately minus 14 mesh and on 35 mesh and a product of relatively fine particle size substantially all minus 35 mesh and predominately minus 65 mesh, and subjecting each of said sized products to separate cleaner steps of pneumatic froth flotation in the presence of a cationic collecting agent formulated to collect the type of silica predominating in each of said sized products.

6. The method of separating silica from a mixture of phosphatic material and silica which comprises subjecting an aqueous pulp of the deslimed and unsized mixture to a first rougher step of pneumatic froth flotation in the presence of a cationic collecting flotation agent and in the course of which finely disseminated air is introduced into a relatively deep column of the aqueous pulp at a plurality of superimposed and vertically spaced levels,

removing a silica froth from the top of the pulp column and a rougher discharging phosphate concentrate in the underflow from the bottom of the pulp column, subjecting said rougher phosphate concentrate to a sizing treatment to obtain a product of relatively coarse particle size and a product of relatively fine particle size, and subjecting each of said sized products to separate cleaner steps of pneumatic froth flotation in the presence of a cationic collecting flotation agent formulated to collect the type of silica predominating in each of said sized products.

7. The method of separating silica from a mixture of phosphatic material and silica which comprises subjecting an aqueous pulp of the deslimed and unsized mixture to pneumatic froth flotation in the presence of a cationic collecting flotation agent and in the course of which finely disseminated air is introduced into a relatively deep column of the aqueous pulp at a plurality of superimposed and vertically spaced levels and water is introduced in a fine state of subdivision into the pulp column at the bottom thereof thereby substantially decreasing the downward velocity of the pulp flow, removing a silica froth from the top of the pulp column and discharging a phosphate concentrate in the underflow from the bottom of the pulp column together with the equivalent of most of the water introduced into the bottom of the pulp column, subjecting said phosphate concentrate to a sizing treatment to obtain a product of relatively coarse particle size and a product of relatively fine particle size, and subjecting each of said sized products to separate cleaner steps of pneumatic froth flotation in the presence of a cationic collecting flotation agent formulated to collect the particular type of silica predominating in each of said sized products, said cleaner steps of pneumatic froth flotation being of the same character as the aforesaid rougher step of froth flotation.

8. The method of separating silica from a mixture of phosphatic material and silica which comprises subjecting an aqueous pulp of the deslimed and unsized mixture to pneumatic froth flotation in the presence of a cationic collecting flotation agent and in the course of which finely disseminated air is introduced into a relatively deep column of the aqueous pulp at a plurality of superimposed and vertically spaced levels and water is introduced in a fine state of subdivision into the pulp column at the bottom thereof thereby substantially decreasing the downward velocity of the pulp flow, removing a silica froth from the top of the pulp column and discharging a phosphate concentrate in the underflow from the bottom of the pulp column together with the equivalent of most of the water introduced into the bottom of the pulp column, subjecting said phosphate concentrate to a sizing treatment to obtain a product of relatively coarse particle size predominately minus 14 mesh and on 35 mesh and a product of relatively fine particle size substantially all minus 35 mesh and predominately minus 65 mesh, and subjecting each of said sized products to separate cleaner steps of pneumatic froth flotation in the presence of a cationic collecting flotation agent formulated to collect the particular type of silica predominating in each of said sized products, said cleaner steps of pneumatic froth flotation being of the same character as the aforesaid rougher step of froth flotation.

**References Cited** in the file of this patent

UNITED STATES PATENTS

| | | |
|---|---|---|
| 2,156,245 | Mead et al. | Apr. 25, 1939 |
| 2,176,107 | Smith | Oct. 17, 1939 |
| 2,185,224 | Ralston | Jan. 2, 1940 |
| 2,357,419 | Mead et al. | Sept. 5, 1944 |
| 2,369,311 | Mead et al. | Feb. 13, 1945 |

OTHER REFERENCES

"Handbook of Mineral Dressing" (Taggart), published by John Wiley and Sons, Incorporated (New York), 1927, pages 12–14 relied on.