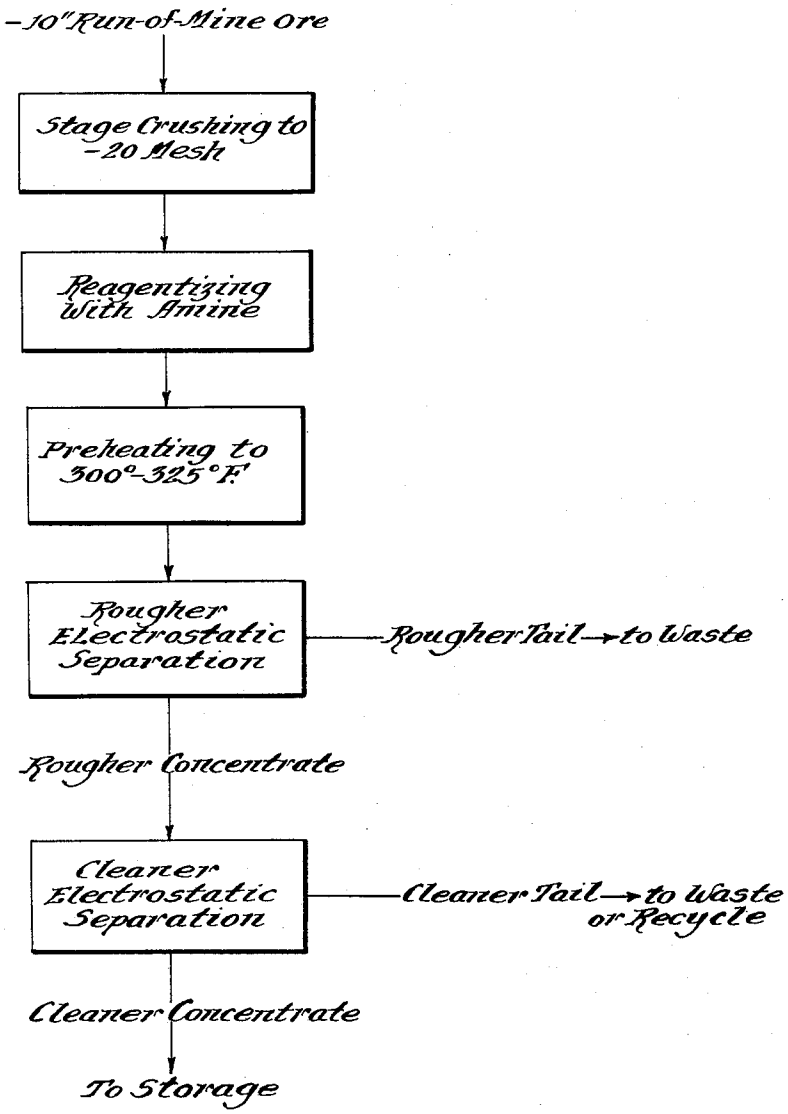
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BENEFICIATION OF ORES
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CALCITIC-PHOSPHATE ORE BENEFICIATION PROCESS FLOWSHEET



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BENEFICIATION OF ORES

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This invention generally relates to the electrostatic beneficiation of minerals and more particularly it relates to the electrostatic beneficiation of calcitic phosphate ores. The novel process of this invention is applicable to the beneficiation of calcite-apatite ores in which these principal minerals may be substantially completely liberated from each other.

Calcite is calcium carbonate, $CaCO_3$; CaO, 56.0%; CO_2, 44.0%. Apatite is essentially a calcium phosphate varying widely in chemical composition, but usually containing Cl, F, and OH in its crystal lattice.

For the major uses of apatite, the mineral is preferably in concentrated form, that is, preferably substantially free of impurities. The phosphate industry requires, for the production of fertilizers, superphosphate and triple superphosphate, a phosphatic material of relatively high BPL (bone phosphate of lime) content and imposes price penalties where impurities are present in excess of certain maximum fixed percentages.

In the Province of Ontario, Canada, large deposits of calcite-apatite occur. Large deposits of such ores are also found in the Rocky Mountains and the Appalachian Mountains, as well as in the Mediterranean area of North Africa where commercial mining and production are practiced. Calcitic phosphate ores are also found in the Scandinavian region.

In order to be attractive on a commercial scale, a process for beneficiating a calcite-apatite ore should produce an apatite concentrate which is substantially free of calcite values. The present invention is directed to providing such a process.

Accordingly, it is an object of the present invention to provide an electrostatic process for beneficiating a calcitic phosphate ore.

It is another object of the invention to provide an electrostatic process for beneficiating a calcite-apatite ore wherein concentrates of commercial grade may be obtained at a high percentage recovery of P_2O_5.

Another object of the invention is to provide an electrostatic method for beneficiating a calcite-apatite ore which, while operable in a series of passes through one or more electrostatic fields to upgrade the P_2O_5 content of the concentrate, is capable, under optimum conditions, of producing a commercially attractive concentrate in a single pass.

An additional object of the invention is to provide an electrostatic process for beneficiating slime-bearing calcite-apatite ores to produce commercially attractive apatite concentrates with a high total P_2O_5 recovery.

These and other objects and advantages of the present invention will be apparent as the description of the invention progresses. The drawing is a diagrammatic flowsheet of an ore beneficiation process referred to in the examples.

In accordance with the present invention, it has been discovered that eminently satisfactory beneficiation of calcite-apatite ores and minerals can be achieved electrostatically by a series of critical and interdependent process steps. Generally described, the present invention is an electrostatic process for beneficiating a calcite-apatite material which comprises treating a substantially dry mixture containing discrete granules of apatite and

calcite with a surface active agent, inducing the treated granules to accept differential charges, and passing the treated and charged granules into an electrostatic field without substantial alteration of their charge to separate a fraction rich in apatite.

Separation of various materials including ores by electrostatic procedures long has been viewed by the art as a desirable expedient both from the standpoint of efficiency and economy. In practice, some success has been achieved in separating mixtures of conductive materials and mixtures of conductive and non-conductive materials. Except in isolated instances, however, it has heretofore been found to be difficult to effect commercially satisfactory separations between essentially non-conductive materials and especially non-conductive materials such as the naturally occurring mixtures of calcite and apatite.

In successful prior art conductivity-type electrostatic separations, advantage has been taken of the differential ability of various conductive materials to dissipate an electrical charge. In the usual conductivity separation of the prior art, the dry, comminuted mixture is fed to the grounded conveyor roll of a roll-type electrostatic separator, and in the course of delivery to the electrostatic field created by a closely spaced, oppositely charged electrode is charged by bombardment with ions, usually in the form of corona discharge. The conductive or more conductive components of the mixture rapidly lose their charge to the conveyor roll and either follow their natural trajectory into the collection zone below or else are horizontally displaced by the attractive and repulsive forces of the electrostatic field. The non-conductive or less conductive components retain their charge longer than the conductors, are attracted by the conveyor roll in varying degree, and either fall from or are scraped from the surface of the conveyor roll at points circumferentially beyond the normal point of gravitational dissociation.

It at once will be recognized that mixtures of essentially non-conductors do not readily lend themselves to separation by the conductivity method described. The art, therefore, has been forced to various expedients in an effort to effect commercially attractive separations of non-conductive materials.

It has been discovered that under certain conditions, particulate non-conductive materials will assume an electric charge by contact electrification. Such charge may be developed by contact with metal, a grounded conductor, or by particle-to-particle contact. The charge developed by an isolated, pure material in contact with a metal or grounded conductor, however, affords no basis for accurate prediction of the type or magnitude of charge which the same particle will assume when contacted with a dissimilar non-conductive particle during intentional agitation or as a result of normal manipulation. When, for example, feldspar and quartz are individually contacted with a metal such as iron, aluminum, or zinc, the feldspar assumes a weakly negative charge while quartz becomes strongly negatively charged. When a comminuted feldspathic ore containing feldspar is charged by particle-to-particle contact, the quartz becomes negatively charged and feldspar assumes a positive charge. On the other hand, when a comminuted phosphate ore containing calcium phosphate, quartz and a small amount of feldspar is charged by particle-to-particle contact, the phosphate assumes a positive charge while the quartz and feldspar charge negatively.

The mere fact that non-conductive mineral substances will assume differential charges regrettably does not permit ready electrostatic separation. Instead, the charge differential must be of sufficient magnitude that an elec-

trostatic field of practical strength will effect the degree of horizontal particles displacement requisite to commercial operations. Accordingly, the art is confronted with the problem of ascertaining conditions under which the required charge differential may be achieved with the recognition that the charging characteristics of any given mineral component of a particular association are often unpredictable.

It previously as been determined that an acceptable separation of calcium phosphate from silica in a phosphate ore may be achieved by subjecting the liberated phosphate ore to a heat treatment wherein the liberated ore is heated to a temperature above 150° F., is then caused to accept differential charges and then passed through an electrostatic field to raise the P_2O_5 content of the concentrate. This process is described in Lawver U.S. Patent No. 2,805,769.

One of the difficulties encountered in effecting an electrostatic beneficiation of phosphate-calcite ores has been the presence of slimes which effectively mask the ability of phosphate and calcite to assume differential electrical charges of the required magnitude. The slimes occurring in most phosphate ores owe their origin to the clay-like substances naturally associated with the ore. Such slimes are removable to a large extent by scrubbing procedures known to the prior art; however, it is difficult and costly to reduce the slime content to very low levels. Moreover, if phosphate ore is scrubbed with water to remove slimes, practical economic considerations dictate that the wet ore be beneficiated by a flotation process rather than subjected to the expensive drying procedures requisite to presenting dry surfaces for differential charging in an electrostatic process. In view of these and other difficulties experienced by the prior art, a primary object of the present invention is to provide a commercially attractive electrostatic method for beneficiating slime-bearing calcite-apatite ores.

Ores which may be beneficiated by the method of this invention are the natural calcite-apatite ores containing other minerals. A large deposit of such an ore is situated near Nemegos, Ontario, Canada, and large deposits are found in the Mediterranean area and the Scandinavian region. The apatite is generally found as fluorapatite and may also be found as chloroapatite, or hydroxyapatite or mixtures of two or more of these species of apatite. In addition to the natural ores, the process of the invention may be used to beneficiate artificially created mixtures or concentrates obtained in various beneficiation processes.

Regardless of the source of the ore, it is necessary that the phosphate values be liberated from the calcite and other gangue components of the mixture and be of the mesh size amenable to electrostatic separation in an electrostatic field of practical strength. Accordingly, the materials to be treated are, where necessary, reduced to a particle size of less than about 10 mesh and preferably to a particle size of less than about 14 mesh. The lower limit of particle size is about 325 mesh and desirably will be about 200 mesh, since —200 mesh material creates a serious dust problem and in time builds up a dust layer on the equipment, including the electrodes. Further, it has been found that different types of separators available vary in their ability to tolerate fines. For example, it is usually undesirable to employ particles smaller than 100–150 mesh with roll-type separators, while the so-called free-fall type separator will effectively separate the smaller particle sizes.

In practically all electrostatic separations, it is necessary for satisfactory results that the surfaces of the particles be substantially dry, thus removing the complicating effects of moisture in the charging and separation steps. Additionally, it has been found that the ability of some mineral substances to assume and retain differential charges is materially affected, inter alia, by temperature, as established by experience of the art in the

electrostatic beneficiation of unreagentized calcite-apatite ores. In the process of the present invention, separations are materially improved by conditioning the comminuted material at a temperature above that necessary merely to obtain dryness. Preferably, temperatures of at least 150° F. are employed and a temperature of at least 150° F. is desirably maintained during the charging and up to the point of introduction into the electrostatic field. Higher temperatures which do not deleteriously affect the mineral can be employed, and, in many instances, are required in order to satisfactorily prepare the particle surfaces for optimum separations. In general, temperatures of between about 200° F. and about 1000° F. produce the desired results. Temperatures within the range of from about 250° F. to about 550° F. have produced good results. Obviously, the use of temperatures higher than those resulting in optimum beneficiation cannot be justified economically.

The surface-active agent or reagent employed as an essential facet of the method of the invention may be added to the comminuted mixture to be separated either before or after the heat conditioning step. If added prior to heating, the conditioning temperature employed must be below the decomposition temperature of the reagent. In any event, the reagent is preferably added to the mixture to be separated while the mixture is in a substantially dry condition in order to obtain the benefits of the invention. Moreover, it is desirable in applying the reagent to the material, to maintain the particulate material in an unagglomerated condition, as by agitation, while bringing the reagent into contact with the particulate material.

The particulate mixture may be conditioned with the surface-active agent in any suitable manner. If the reagent is normally liquid, is molten, or is in solution or suspension, it may be poured or sprayed over the particles either while the particles are static or are under agitation. Similarly, the particulate material may be suitably treated with vaporized reagent. Solid reagents may be poured or sifted on the particles. Temperature and/or agitation then may be employed to produce the desired dry surfaces.

The reagent employed in the process of the invention may be selected from a variety of surface-active materials known to the art. The specifically preferred reagents are of the long-chain aliphatic amines, preferably containing from 8 to 20 carbon atoms in the molecule and more preferably from 12 to 20 carbon atoms in the molecule, such as octadecyl amine, hexadecyl amine, dodecyl amine, cetyl amine and the tall oil, coconut oil and soya oil amines. The amines have given excellent results when used in the process of this invention. Amine salts of carboxylic acids such as rosin amine acetate advantageously also may be employed. Sulfated alcohols are also effective surface-active agents. Sulfonates such as the petroleum sulfonates, sodium keryl benzene sulfonate, sodium polypropylene benzene sulfonate, sodium lauryl sulfonate, and the like, may be employed as surface-active agents, as may the natural resin acids such as abietic, dihydroabietic, pimaric acids; sulfonated and sulfated fatty acids, and the like. Carboxylic acid esters such as ethyl hexyl acetate and fatty acid soaps such as the alkali and alkaline earth metal soaps of stearic, lauric, oleic, and palmitic acids may also be used. The amines are, however, specifically preferred, since it has been determined that their use gives particularly good results in the electrostatic beneficiation.

The reagents are employed in small proportions. Generally, the amount of reagent employed will be within the range of from about 0.01 to about 10.0 pounds of surface-active agent per ton of the particulate mixture to be treated and separated in accordance with the method of the invention. With calcite-apatite ores from the Virkby, Finland and the Nemegos, Ontario, Canada areas,

from about 0.2 to about 2.5 pounds of amine per ton of particulate mixture is preferably employed, and more preferably from about 0.5 to about 1.0 pound of amine per ton of particulate mixture is employed. As indicated, the normally semi-solid reagents may be applied in solution or suspension. Since the reagents are employed in small proportions, it frequently is desirable to use an extender such as fuel oil, kerosene, and the like.

In accordance with the present invention, the reagentized particles are induced to assume differential charges by the mechanism of contact electrification. Contact electrification results from the movement of charged particles in response to such stimuli as differences in escape rate of positive and negative charges, or transfer of electrons or ions across an interface due to differences in energy levels and the like. It has been determined that real crystals never attain the static perfection of an ideal crystal lattice and that a real crystal may have distorted surfaces, displaced ions or atoms, interstitial sites and surface sites, and charge displacement due to separated anion-cation pairs of abnormal ionized atoms and trapped electrons. It is postulated that these traps are capable of acting as donors and acceptors of electrons, and frequently it is these traps that are probably the controlling influences in contact electrification of minerals.

Charging by contact electrification normally is obtained by one of two mechanisms, viz., contact with a metal or grounded metal surface or by particle-to-particle contact. When nonconductive particles such as the particles in a liberated phosphate ore contact a metal or grounded metal plate, the particles normally are charged negatively, but at different magnitudes of charge. When the difference in magnitude is great enough, effective separations are possible. When charging is effected by particle-to-particle contact under conditions present in the method of the invention, the particles of phosphate and calcite are charged to opposite polarity and separation is more readily effected. While a relatively small amount of the charging obtained in the process of the invention results from particle-to-metal equipment contact, most of the charging is effected by the more desirable particle-to-particle contact, either as a result of intentional agitation or agitation incident to handling of the feed material during and after the heating and reagentizing steps. The sign of the surface charge to be expected in particle-to-particle contact electrification depends on the probability of the particle making contact with surface A, B, C, etc., and the relation of the surface energies that control the sign of contact electrification of the particles against A, B, C, etc.

It has been discovered that greatly improved differential charging of the particles is accomplished in accordance with the invention by essentially particle-to-particle contact while the dry comminuted material is maintained at a temperature of at least 100° F. Ideally, the particles would not contact a metal or grounded metal surface during the charging operation, since as previously indicated, contact with grounded metal surfaces usually causes all dry particles to accept a negative charge. On the other hand, where the charging of the particles is accomplished essentially by particle-to-particle contact, the surface charge found on two individual mineral species in the ore is equal and opposite in sign. Accordingly, the charged particles move in opposite directions in an electrostatic field. Thus, in the process of the invention, it becomes possible effectively to separate nonconductive particles.

The desired particle-to-particle charging may be effected in numerous ways, such as by tumbling the particles in a revolving drum or down an elongated chute in such quantity that contact between the particles and the chute is at a minimum. Alternatively, the comminuted mineral, while maintained at the proper temperature, may be delivered from the drying apparatus to the electrostatic separator by means of a vibrating trough. At high throughput, the great preponderance of charging is engendered by particle-to-particle contact rather than by contact of the particles with the apparatus. Suitable charging also may be obtained by air agitation of the hot comminuted mineral.

Following the preliminary heating, reagentizing and differential charging procedures, the particulate mixture then is passed into a suitable electrostatic field for separation. At the time of entry into the electrostatic field, the treated material should be at a temperature of at least about 100° F. and preferably at a temperature within the range of from about 140° F. to about 350° F., although a higher temperature may be employed as previously indicated. Because of the nature of the treatment and charging of the material, the phosphate particles and calcite particles are strongly charged to opposite polarity with the result that a commercially attractive separation often may be achieved in a single pass through an electrostatic field.

As long as the process limitations hereinbefore delineated are observed, the type of separation apparatus employed is not critical, the only limiting factor in terms of apparatus being that the charges on the differentially charged particles be substantially unaltered during delivery to and passage through the electrostatic field. Accordingly, apparatus with which a material amount of charging by inductive conduction is obtained is not desirably employed.

The so-called free-fall type of separator is preferred, inter alia, (*a*) because the usually employed elongated, vertically disposed electrodes provide longer residence times in the field, (*b*) because finer particles may be employed than with roll-type equipment, and (*c*) because the apparatus is less expensive and more easily serviced. However, excellent separations may be achieved with roll-type apparatus wherein the conveyor roll is employed merely as a means of delivering the differentially charged particles to the electrostatic field and substantial charging by inductive conduction is avoided. Suitable free-fall type apparatus is disclosed in U.S. Patent No. 2,782,923 to Charles C. Cook et al. Suitable roll-type apparatus is disclosed in Taggart, "Handbook of Mineral Dressing", 1956, chapter 13, such roll-type apparatus being operated to prevent substantial charging by inductive conduction.

The strength of the electrostatic field which will effectively alter the normal trajectory of ore particles depends on the mass of the particle and the total surface charge on the particle. The potential gradient desirably will vary from about 1,000 volts to about 5,000 volts per inch of distance between electrodes in separating material of relatively fine particle size, and from about 3,000 volts to about 15,000 volts per inch for beneficiating coarser particles. In all such discussion of field strength, it must be borne in mind that corona discharges which ionize air are to be avoided. With free-fall apparatus, it generally is preferred to operate with a total impressed difference of potential in the range of about 30,000 volts to about 250,000 volts, while with a roll-type separator, a range of about 10,000 volts to about 75,000 volts is preferred. This voltage difference should be maintained by means of a direct current potential source substantially free of ripple. A steady supply of direct current may be obtained with inexpensive filtering apparatus by the use of such equipment as a rectified radio frequency power supply.

Where ore particles are subjected to a series of separations, the feed to subsequent stages often will exhibit progressively reduced response to the electrostatic fields. This reduced response often may be due to loss or leakage of charges from the ground or particles or coating of the charged particles with fines. Such weak-responding concentrates may be restored or reactivated by passage through an impactor to create new surfaces and again recharging by frictional or other methods that give rise to differential electrification, which recharging may include a reheating in accordance with the treatment hereinabove described. Where a plurality of separation stages is employed, it is also within the scope of the in-

vention to re-reagentize the granular ore particles between stages.

When a satisfactory beneficiation is not accomplished in a single stage of electrostatic separation, the usual procedure is to separate a concentrate in a so-called rougher stage and to upgrade the concentrate by treatment in one or more so-called cleaner electrostatic separation stages. While the breadth of the range of temperature at the time of passage through the electrostatic field appears adequate to allow for cooling during passage through the stages necessary to make the desired grade of product, it frequently happens that this is not the case. One of the primary reasons for this failure is that in order to reduce the number of separation stages, it is desirable to make the first or rougher separation at or near the temperature of about 175° F. to about 200° F., depending on the character of the ore. Concentrate from the rougher separation thus will cool more or less rapidly depending upon the difference in temperature between 200° F. and the surrounding atmospheric temperature. After passage through one or more concentrate upgrading stages, it is found that sometimes the material has cooled below a temperature at which a measurable degree of upgrading will occur. When a phosphate-calcite ore has become too cool, or picked up too much surface moisture, it sometimes happens that neither the concentrate nor the tail product will respond to further passes through electrostatic fields of the same, lower, or higher potential gradient.

Cooling conditions at times may be such that with some ores requiring a plurality of passes, high P_2O_5 products can be secured in three consecutive quick passes through electrostatic fields without reheating the solids. On the other hand, cooling of solids as during winter seasons may be so fast that precautions must be taken in handling of a rougher concentrate to obtain satisfactory separation in a first cleaner stage without reheating between the rougher and first cleaner operation. When conditions prevail such that substantial cooling of the ore particles takes place during the operation, reheating is found to be beneficial in order to make products of acceptable commercial grade.

In general, it has been found that where additional passes through an electrostatic field are desirable, a secondary heat treatment whereby the temperature of the solids is maintained at, or is raised to or above 200° F. between separation stages following the first or rougher separation, not only produces products meeting grade specifications, but also reduces the number of separation stages to obtain products meeting such grade specifications. Optimum conditions, of course, exist for various ores and for the various types of electrostatic separators and readily may be determined.

When the material to be separated passes through a series of electrostatic fields, the preferred mode of operation provides for the collection of three fractions from each electrostatic field. How these fractions are further treated depends upon whether the emphasis is on a rejection of a relatively pure tail, recovery of a relatively pure concentrate, or both. For example, if emphasis is on the concentrate fraction, the operation of the first or rougher separation stage may be such that a throwaway tail is taken in this very first step. Under such circumstances, the rougher concentrate fraction is treated in one or more cleaner sections, in which event the tail product from the cleaner section is either a throwaway material or is recycled to a point where the composition of the material corresponds roughly to the composition of the feed material to a separation unit. In an alternative mode of operation, the rougher concentrate and rougher tail are not final products, and accordingly are subjected to one or more stages of separation to segregate, for example, apatite from calcite. In some instances a middling fraction is recovered during rougher, scavenger, or cleaner pass separation and is recycled either to the feed unit in which it is prepared, or to a point where the composition of the middling corresponds roughly to the composition of the feed material to a separation unit. It will be apparent that many varations may be made in the manipulative steps of the process where it becomes desirable or necessary to subject a particular feed to a plurality of stages of electrostatic separation.

Having generally described the method of the invention, the following examples are given to illustrate specific embodiments thereof:

Example I

A Finnish calcitic phosphate ore had the following mineralogical composition:

Mineral:	Assay, percent
Apatite	25
Calcite	64
Dolomite	11
Fe-oxides and others	Trace
	100

and the following chemical analysis:

	Percent
BPL	22.29
Insolubles	0.15
Fe_2O_3	0.90
Al_2O_3	0.07
CO_2	33.87
F	0.56
MgO	2.12

The ore was comminuted and screened to produce a fraction containing —20 mesh particles. Another sample of the ore was comminuted and screened to produce a —28 mesh fraction. A sample of the —28 mesh fraction was tumbled in a metal container for five minutes with 0.6 pound of amine flotation reagent, sold by Armour and Company under the trade name Armac T, per ton of ore, dissolved in twice its weight of kerosene (1.2 pounds of kerosene per ton equivalent). The kerosene served as a dispersant. The reagentized fraction was heated at approximately 300° F. for one hour, and prior to any substantial cooling, was passed in a layer of between ⅛ inch and about ½ inch in depth through a vibrating trough. The hot, differentially charged material was then dropped as freely falling bodies through a free-fall type electrostatic separator. The temperature of the ore during the drop was approximately 200° F. The field was maintained between the spaced vertical electrodes of the separator at a gradient of approximately 7,500 volts per inch. The electrodes were 10 inches apart with the total impressed voltage being 75,000 volts. Below the electrodes, seven plans were properly arranged so as to collect all the material passing between the electrodes as seven separate fractions.

The results of the single pass separations were as follows:

Pan	Percent weight	Percent BPL	Percent dist. BPL
1	12.8	70.58	42.3
2	15.1	59.43	42.1
3	7.4	30.92	10.7
4	12.2	5.46	3.1
5	18.8	1.04	0.9
6	20.7	0.60	0.5
7	12.0	0.76	0.4
Composite	100.0	21.34	100.0

A fraction of the —20 mesh fraction and a fraction of the —28 mesh fraction, without reagent added, were also separately heated and subjected to charging and electrostatic separation under substantially the same conditions as those employed for the reagentized fraction.

The results of the single pass separation were as follows:

Pan	—20 mesh ore			Pan	—28 mesh ore		
	Percent weight	Percent BPL	Percent dist. BPL		Percent weight	Percent BPL	Percent dist. BPL
1-2	13.1	59.00	35.4	1-2	14.9	68.39	46.9
3	12.7	41.95	24.4	3	11.1	46.98	24.1
4	29.6	21.20	28.8	4	23.3	19.88	21.3
5	28.6	5.73	7.5	5	28.8	3.22	4.3
6	11.0	3.93	2.0	6	16.0	2.56	1.9
7	5.0	8.41	1.9	7	5.9	5.68	1.5
Comp.	100.0	21.82	100.0	Comp.	100.0	21.71	100.0

By comparing the results from separating the reagentized and unreagentized —28 mesh fractions, it will be noted that conditioning the ore with the amine considerably increased the degree of separation. It may also be noted by comparing the results from the runs on the unreagentized fraction that the —28 mesh ore was found to be more readily upgraded than was the —20 mesh ore.

Example II

Another sample of the —28 mesh fraction was subjected to electrostatic separation in substantially the same manner and under the same conditions as those employed on the reagentized ore in Example I, except that 0.8 pound of amine flotation reagent per ton of ore (dissolved in 1.6 pounds of kerosene per ton of ore) was employed. Results were as follows:

Pan	Percent weight	Percent BPL	Percent dist. BPL
1	13.2	75.28	46.6
2	9.5	61.07	27.2
3	9.0	47.96	20.2
4	9.1	6.91	2.9
5	19.1	2.02	1.9
6	24.6	0.63	0.7
7	15.5	0.74	0.5
Composite	100.0	21.34	100.0

Example III

The procedure of Example II was repeated on a sample of the —20 mesh fraction. Results were as follows:

Pan	Percent weight	Percent BPL	Percent dist. BPL
1	10.0	78.23	36.7
2	8.4	74.07	29.3
3	6.9	63.58	20.6
4	11.3	19.55	10.3
5	28.8	1.50	2.0
6	23.2	0.54	0.6
7	11.4	0.82	0.5
Composite	100.0	21.29	100.0

Comparing the results of Example II with the results of Example III, it may be noted that the conditioned —20 mesh material responded better than the conditioned —28 mesh material.

The results of the tests of the above examples establish (1) that a satisfactory separation of apatite from calcite can be achieved in accordance with the invention in a single pass in a free-fall process; (2) that since —20 mesh ore conditioned with an aliphatic amine responds better than —28 mesh conditioned or unconditioned ore considerable savings in grinding costs may be realized.

Example IV

In order to further illustrate the advantages of using amine-reagentized feed, the following comparative tests were performed. A sample of the —28 mesh ore described in Example I with no reagent treatment and a sample of the —20 mesh ore described in Example I after reagentizing with 0.8 pound per ton of Armac T and 1.6 pounds per ton of kerosene were each subjected to electrostatic beneficiation using the flowsheet presented in the drawing. Except as noted, the conditions were substantially the same as described in Example I. Maximum BPL recovery at an 80% BPL concentrate grade was desired for the process to be attractive commercially.

Results using the non-reagentized —28 mesh ore were as follows:

Product	Percent weight	Assay, percent		Percent distribution of BPL
		BPL	CO_2	
Cleaner concentrate	14.1	80.0	4.6	53.5
Cleaner tail	40.1	20.2	-------	38.4
Rougher tail	45.8	3.7	-------	8.1
Composite	100.0	21.1	33.9	100.0

A much greater recovery of 80% BPL concentrate was obtained using the amine-reagentized —20 mesh ore as shown below:

Product	Percent weight	Assay, percent		Percent distribution of BPL
		BPL	CO_2	
Cleaner concentrate	23.2	80.0	4.6	87.1
Cleaner tail	13.4	15.5	-------	9.8
Rougher tail	63.4	1.0	-------	3.1
Composite	100.0	21.3	21.3	100.0

Example V

A complex, low-grade calcitic phosphate ore from Nemegos, Ontario, Canada, had the following mineral composition:

Mineral:	Assay, percent
Apatite	11.0
Calcite	77.0
Pyroxene	6.5
Magnetite	3.0
Feldspar	1.0
Pyrochlore	0.5
Pyrite	0.5
Biotite and quartz	0.5
	100.0

and the following chemical analysis:

	Percent
BPL	11.1
CO_2	26.9
Nb	0.1

A representative sample of the ore was stage crushed to —24 mesh, dedusted at 150 mesh, and subjected to high-intensity dry magnetic separation to remove most of the magnetic material, namely, pyroxene, magnetite, biotite, and some of the pyrochlore and pyrite. The nonmagnetic fraction, containing primarily apatite and calcite, was divided into two fractions. One fraction was heated to 325° F. and subjected to a rougher-cleaner pass electrostatic separation as is described in Example IV. The second fraction was tumbled for 3 minutes with 0.6 pound per ton of an amine reagent sold by Armour & Co. under the tradename Armac OD, dissolved in twice its weight of kerosene (1.2 pounds of kerosene per ton equivalent). The reagentized ore was then heated to 325° F. and subjected to a rougher-cleaner separation at substantially the same conditions as the first fraction.

Using the amine reagentized feed, superior results were obtained as shown by the following data:

Pan	Using no reagent			Using 0.6 lb./ton Armac OD		
	Percent weight	Percent BPL	Percent dist. BPL	Percent weight	Percent BPL	Percent dist. BPL
Cleaner pass:						
1–2	7.0	68.2	35.8	4.9	79.4	26.3
3	3.3	66.0	16.4	7.1	78.1	37.5
4	4.7	55.2	19.4	7.1	51.2	24.6
5	3.8	32.0	9.2	3.7	9.2	2.3
6–7	2.8	22.8	4.8	4.4	4.2	1.2
Rougher pass: 4, 5, 6–7	78.4	2.5	14.4	72.8	1.6	8.0
Composite	100.0	13.3	100.0	100.0	14.8	100.0

The above examples establish that calcitic phosphate ores effectively may be beneficiated by electrostatic means when reagentized and processed in accordance with the present invention.

Since modification of specific process details within the scope of the invention will become apparent to those skilled in the art, it is intended that the invention be limited only by the scope of the appended claims.

This application is a continuation-in-part of copending application Serial No. 758,922, filed September 4, 1958.

I claim as my invention:

1. A method of beneficiating calcitic phosphate materials which comprises treating a substantially dry mixture containing discrete granules of calcite and phosphate having a particle size of less than about 10 mesh with a cationic surface active agent selected from the group consisting of long chain aliphatic amines and amine salts of carboxylic acids, inducing the treated granules to accept differential charges by contact electrification, and passing the treated and charged granules into an electrostatic field without substantial alteration of the charge to separate a fraction rich in phosphate.

2. A method of beneficiating calcitic phosphate materials which comprises heating a mixture containing discrete granules of calcite and phosphate materials having a particle size less than about 10 mesh and a temperature of at least about 100° F., treating the conditioned mixture while substantially dry and at a temperature of at least about 100° F. with a cationic surface active agent selected from the group consisting of long chain aliphatic amines and amine salts of carboxylic acids, inducing the treated granules to accept differential charges by contact electrification, and passing the treated and charged granules while at a temperature of at least about 100° F. into an electrostatic field without substantial alteration of the charge to separate a fraction rich in phosphate.

3. A method of beneficiating an ore containing calcite and apatite which comprises comminuting the ore to a particle size at which the apatite values are substantially completely liberated from the calcite and other gangue materials and the particle size of the comminuted ore is less than about 10 mesh, heating the conditioned ore at a temperature of at least 100° F., treating the conditioned ore while substantially dry while at a temperature of at least 100° F. with a cationic surface active agent selected from the group consisting of long chain aliphatic amines and salts of carboxylic acids, inducing the treated ore while at a temperature of at least 100° F. to assume differential charges by contact electrification, and passing the treated and differentially charged ore while at a temperature of at least 100° F. into an electrostatic field without substantial alteration of the charge on the particles to separate a fraction rich in apatite and a fraction rich in calcite.

4. A method according to claim 3 in which the aliphatic amine contains from about 12 to about 20 carbon atoms in the molecule.

5. A method according to claim 3 in which the surface active agent is employed in the range of from about 0.01 to about 10 pounds per ton of ore.

6. A method according to claim 3 in which the particles are charged by essentially particle-to-particle contact.

7. A method according to claim 1 in which said cationic surface-active agent is used with an extender.

8. A method according to claim 3 in which said cationic surface-active agent is extended with kerosene.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,063,561 November 13, 1962

Robert E. Snow

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 9, for "as" read -- has --; column 12, line 17, after "and" insert -- amine --.

Signed and sealed this 7th day of May 1963.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents