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METHOD OF FRACTIONALLY SEPARATING PULVERULENT MATERIALS

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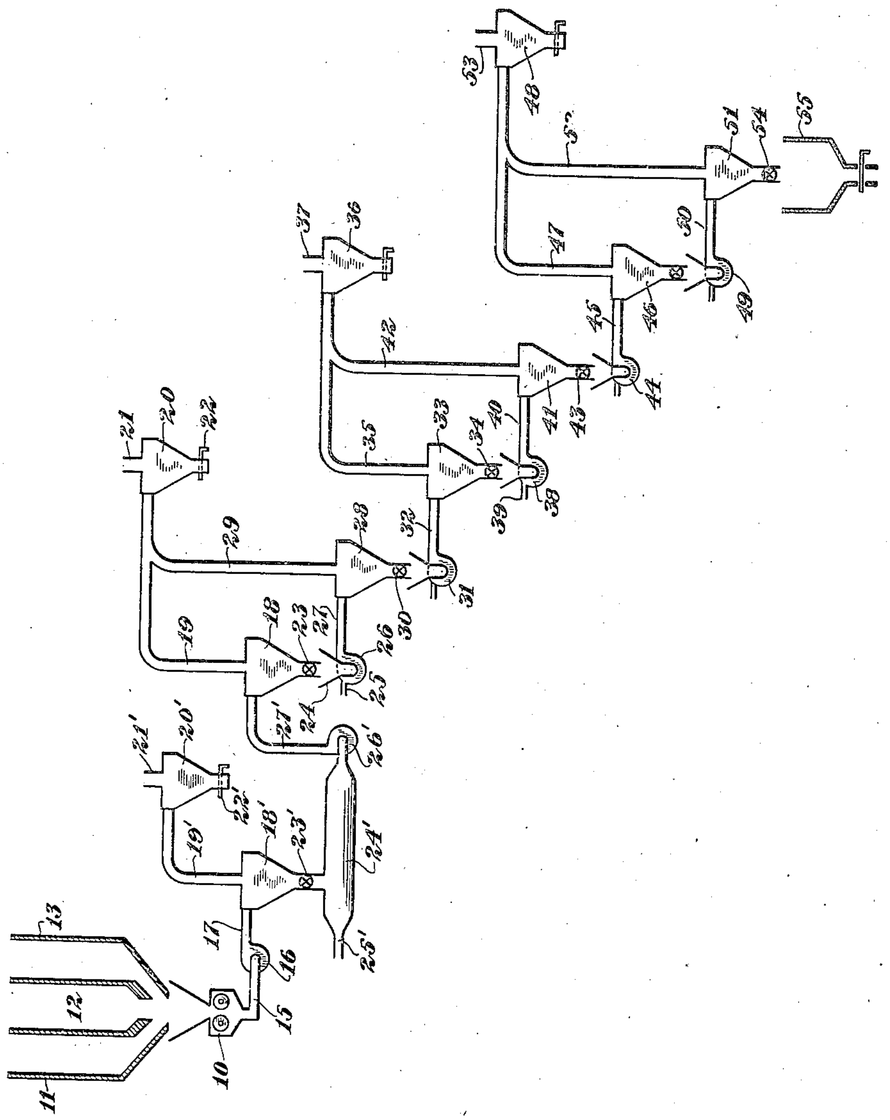

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METHOD OF FRACTIONALLY SEPARATING PULVERULENT MATERIALS

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This invention relates to a method of treating materials consisting of a heterogeneous mixture of particles of various sizes and of different physical characteristics, whereby two or more products in finely divided form may be obtained, each of said products containing particles of more homogeneous particle size and of particular physical characteristics. The invention also relates to a form of apparatus in which the method may be put in operation.

The invention is adapted to the treatment of inorganic or mineral materials in which the material is milled, disintegrated or pulverized so as to produce a finely divided pulverulent or granular material containing particles of various sizes, and in varying amounts.

The invention is particularly adapted to the production of improved types of filter aids and other powdered products from diatomaceous earth or to other finely divided or pulverulent materials which contain components or particular particles having highly desirable characteristics, which render them of great value when isolated from the other particles with which they are intimately mixed.

An object of this invention is to disclose and provide a new and improved method of selectively classifying a material containing a heterogeneous mixture of particles of various sizes and of differing physical characteristics into component groups, each of said component groups comprising particles of similar size or characteristics.

Another object of this invention is to disclose and provide a method of fractionally separating a finely divided or pulverulent material containing particles of various sizes, into components which contain particles of more homogeneous size.

Another object is to disclose and provide a method whereby a finely divided or pulverulent material containing a plurality of components or component groups may be separated into such components or component groups, with accuracy.

More specifically, an object of this invention is to disclose and provide a method of treating diatomaceous earth whereby various finely divided components may be fractionally separated therefrom, such components having particularly desirable characteristics which are not evidenced when such mineral material is merely reduced to finely divided form in accordance with prior methods.

A still further object of this invention is to disclose and provide means particularly adapted to fractional selective classification of finely divided or pulverulent materials into component groups.

Although other finely divided and pulverulent materials may be treated in accordance with this invention, the subsequent detailed description will be particularly directed to the specific and preferred embodiment of the invention that is the treatment of a diatomaceous earth. Reference will be had to the appended drawing, which diagrammatically illustrates apparatus which may be employed in carrying out the invention.

In general, the method of this invention comprises the following steps and conditions. The material to be treated is first preferably milled, pulverized or disintegrated, such reduction taking place in equipment adapted to carry out the operation with least injury to the desirable components of the material. Such reduction, however, should be thorough.

It is desirable that the diatomaceous earth be reduced to discrete particles, i. e., each diatom should be separated from its neighbors and all aggregates broken up. Such milling, however, should not rupture or break any large proportion of the diatom, but should be directed toward destruction of the reticulate or continuous bond which holds the diatoms in the form of a coherent mass. Such reticulate or continuous bond may be argillaceous, calcareous or siliceous in character, depending on the character of the diatomaceous earth being treated.

The purpose of all milling steps in treatment of diatomaceous earth should be directed toward the removal of the adherent non-diatomaceous or very fine diatomaceous material which is present in the earth.

The milled material is then dried. It is to be understood that, although the material is preferably dried after milling and before selective and fractional classification, this is not to be strictly interpreted, inasmuch as the drying operation may occur in a series of steps interspersed with progressive milling steps. If a gas is used during the subsequent fractional classification, a certain minor drying may even take place during classification or separation.

The method of fractional separation as defined herein may be carried out by employing any fluid, whether gaseous or liquid, provided it does not exert an undesirable effect upon the material treated. Air or waste gases are preferably used; water or other liquids may be employed, but certain difficulties are encountered which are obviated by using gases. When gases are employed during the fractional classification, then it is desirable that the finely divided material fed into the separating system be substantially dry, i. e., not contain more than about 10% of moisture.

The preferably dry, milled material containing the various components is then suspended in a fluid (preferably gaseous) and conveyed by such fluid to a separating zone, where a portion of one component together with the gaseous fluid is removed from the material. The remaining material is resuspended in a fluid and again sent to a separating zone, where another portion of the same component, together with the fluid is removed. These steps are repeated a number of times sufficient to remove the component and similar steps later carried out on the residual material so as to selectively remove another component in fractional increments.

Such removal of a component from a mixture of components is an important feature of this invention. A mixture of components may thus be separated into two or three or as many as eight components. If an attempt is made to remove one component bodily from a mixture in one operation, then it will be found that the attempted separation is not clean cut, that is, either other components are removed together with the desired component, or portions of the desired component are left in the residual material. Diatomaceous earth has been found to be especially difficult to classify into components of substantially homogeneous characteristics due probably to the strong tendency of the particles to adhere to each other. For this reason various attempts to classify diatomaceous earth were finally abandoned for lack of success. These attempts were attended with especially poor results in the cases wherein it was attempted to effect classification by air separation methods.

In addition to the steps described, the invention contemplates mildly milling the material between fractional separations, such mild milling being of importance as it removes smaller particles of components from the larger particles or components, thereby insuring removal of all finer or smaller components from the system. The particular sequence of steps of milling, separating and then further milling is also of importance because of the more efficient results with respect to both milling and separating thus attained. A large amount of fines present during the milling operation tends to protect the larger aggregates from the milling action and this effect is largely avoided by the sequence mentioned.

Such mild milling removes adhering non-diatomaceous material from the larger diatoms, and as a result the final components separated from the heterogeneous mixture are perfectly clean. The importance of producing such clean separations will be explained in greater detail hereinafter.

Furthermore, any aggregates which by chance were left in the material and were not broken up by the initial milling operations are broken up by the subsequent mild milling during fractional separation. As a result, the finished materials obtained by separation in accordance with this invention are clean and of a substantially homogeneous particle size.

Referring to the drawing, the crude material, say for example, diatomaceous earth, may be supplied to a suitable mill 10 from a bin or bins 11, 12 and 13. A plurality of bins may be employed when it is desired to blend certain grades of diatomaceous earth together, although it is to be understood that such blending is not essential to this invention. The mill 10 may be of any desired type, but preferably is of a swing hammer, impact or cage type.

The material milled at 10 or in a plurality of mills such as the mill 10, is preferably reduced to a fine state of division, that is, the material is to be disintegrated so as to break up aggregates and liberate those particles or components which will be subsequently classified. If desired, the milled material may be conveyed by an aeriform current through a conduit 15 to a milling type of fan 16, the discharge conduit 17 leading from such fan to a separating zone such as a cyclone type of air separator 18'.

The fan 16 is preferably of the centrifugal type having blades in the form of paddles of such size with respect to the cross section of the fan housing, as to mill the material. The cyclone separator 18' is preferably designed to collect and retain substantially all of the milled material supplied thereto by the line 17. Air or other gas drawn through the mill 10 by the suction of fan 16 is discharged through the outlet pipe 19' of the separator 18', such air or gas carrying with it the dust-like or extremely fine material. Such dust may be discharged into the atmosphere or it may be conveyed by the line 19' into a separating zone such as the cyclone 20' and deposited therein, while only the air or gas is discharged through the outlet 21'. Instead of using a cyclone 20', any other suitable collecting device such as a bag collector or settling chamber may be employed.

It is to be understood that conditions of low air velocity and low material concentration, but as high a material concentration as is possible to employ and still produce a desired separation, is employed in the system, which includes lines 15, 17, and 19 as well as the fan 16 and cyclones 18' and 20'.

The component removed from the milled material through line 19' preferably consists of the non-diatomaceous or very fine diatomaceous material constituting the reticular or continuous bond in crude diatomaceous earth. Such dust is preferably of the order of one micron and smaller in size, and preferably the dimensions of the conduits, fan speeds and air volumes and velocities are so adjusted that the component separated from the material through line 19' is of the order of one-half micron in size.

This fine component may be discharged from the separator 20' through a valved outlet 22' and stored for use as a slip clay, paint ingredient or other uses.

The major portion of the material retained within the separator 18' may be discharged by a valved outlet 23' into a conduit 24' supplied with heat from any suitable source, such as for example, a separate furnace or the conduit 24' may be supplied with waste gases from a boiler or other source of heat. The heat inlet is indicated in the drawing at 25'. The material fed into the conduit 24' through the valve 23' is suspended in the heated gases and conveyed thereby, with the assistance of a fan 26' and a conduit 27', into a separating zone 18 provided with a discharge line 19 leading to a separation zone such as a cyclone 20.

An outlet 21 may be provided for the discharge of air or gas, whereas the increment or component separated from the milled material and retained by the separator 20 may be discharged through a valved outlet 22.

The material retained within the separator 18 may be discharged through a valved outlet 23 into a hopper or inlet 24 leading to a fan 26. A separate air inlet 25 may be provided for the fan 26. The material fed to the hopper 24 may be slightly milled by the fan 26, such milling being sufficient only to remove any fine non-diatomaceous material adhering to the diatomaceous structure. The material is thus suspended in an aeriform current and discharged by the fan 26 by conduit 27 into a separator 28.

The air velocity is so regulated that another increment of the component removed in separator 18 is also removed in the separator 28 and passes out of the separation zone 28 by line 29, which as indicated in the drawing may connect the same separation zone, namely, the cyclone 20. If desired, the line 29 may be connected with a separate separation zone in which the aeriform current is purified and the increment carried therein removed from the air. The material remaining in cyclone 28 is again discharged through a valved outlet 30 into still another fan 31, and again resuspended in a current of air or other gas and conveyed by discharge line 32 into a separator zone 33.

The major portion of the material is retained within the separator zone 33 and discharged through the valved outlet 34, while the air or gas carrying a part of another component in suspension is discharged from the separation zone 33 by line 35 into a collecting apparatus such as the cyclone 36. The material carried in suspension and sent to the separation zone 36 is retained therein, whereas the air or gas is discharged through the outlet 37.

The major portion of material which has been removed from the aeriform current and remains in the separator 33 is discharged through the valved outlet 34 into the fan 38, provided with air or gas through an inlet 39 and sent by conduit 40 into still another separation zone 41 in which the major portion of the material is removed from the current of air or gas, while another increment of the desired component is removed or separated from the major portion of the material by being permitted to remain in suspension and be discharged from the separator zone 41 by line 42, which is shown in the drawing, leads to the inlet of the separator 36.

The residual material left within the separator 41 is discharged by a valved outlet 43 into still another fan 44 and conveyed thereby through a conduit 45 into a separator or cyclone 46 wherein the major portion of the material is again removed from the aeriform current, and such aeriform current together with a portion of another component is discharged through line 47 into a baghouse or other suitable type of collector such as the cyclone 48.

The major portion of the material remaining in the separator 46 is then fed into a fan 49 and conveyed thereby through a conduit 50 into a cyclone or separator 51. A portion of the component, together with a current of air is discharged from the separator 51 through line 52, said line leading to a suitable air separator in which that component or portion thereof may be separated from the air current. For purposes of simplification, the line 52 is shown leading to the cyclone or separator 48, the air being discharged from such separator by line 53 into the atmosphere.

If we assume that the diatomaceous material being treated consists essentially of four desirable components, it will be seen that the material discharged from the valved outlet 54 of the cyclone or separator 51 into the hopper receiving bin, bagging device or the like 55, represents one component, whereas the materials retained in and discharged by the cyclones 20, 36, and 48 represent three other components.

In the system described hereinabove, the components removed from the original material by cyclones 20, 36 and 48 have been removed in increments, each one of said components requiring two separations or two increments. For example, the material collected in the cyclone 20 has been removed from the major portion of the material in the cyclone 18 and in the cyclone 28, whereas the material collected in separator 36 has been removed in two increments by separators 33 and 41.

It is to be understood that two separations for each component are diagrammatically illustrated on the appended drawing, merely for illustrative purposes and represent the simplest embodiment of this invention.

It will be readily understood that each of the components removed may be removed in three or four increments if desired. The greater the number of increments in which the removal of a component from the major portion of the material is carried out, the cleaner and sharper the separation will be. It is impossible to remove a component of a particular particle size from a heterogeneous mixture of particle sizes in one separation, but such removal can be commercially obtained by utilizing a removal in two steps or in two or more increments in much the manner described and illustrated hereinabove.

Heretofore, it has been thought impossible to repeatedly mill diatomacous earth without breaking the desirable diatomaceous structure. It has been found, however, that a delicate material such as diatomaceous earth, for example, can be repeatedly passed through centrifugal fans a great number of times without breaking up the diatomaceous structure itself, and instead such repeated passage through fans produces a highly desirable purification of the diatoms.

Such purification consists of the removal of adhering diatomaceous and non-diatomaceous, finely divided argillaceous, siliceous or calcareous matter which normally masks the diatoms. Normally, such siliceous, calcareous and argillaceous adhering material interferes with the characteristics of the diatomaceous products in that when a normally milled diatomaceous earth is used for filtration purposes, such adhering non-diatomaceous material is separated from the diatom structure by the action of the liquid in which the filter aid is used, thus forming a suspension in the liquid, such suspension containing high proportions of extremely fine particles which retard instead of assist filtration.

As shown in the drawing, the original material was milled in 10, passed through a cyclone 18′ and dried in conduit 24. The elements marked with the primed numerals may be eliminated from the system, and line 17 from fan 16 may be directly connected to the inlet of separator 18. The drying units and related parts may thus be eliminated whenever the materials fed into the fan 16 are sufficiently dry to be effectively separated.

As stated hereinabove, the moisture content of diatomaceous earth to be fractionally classified in accordance with this invention should not exceed about 10%, and preferably the moisture content should be about 3% or 4%. The presence of moisture is detrimental to accurate classification, inasmuch as aggregates are readily formed.

If the drying unit, including cyclone 18', conduit 24' and fan 26' is eliminated from the apparatus shown on the attached drawing, it will be seen that the major portion of the material being classified passes through six successive air separations. This is subtantially the minimum number of separations which can be employed in fractionally classifying a heterogeneous material into four components, or component groups of substantially homogeneous particle size. In actual practice, the number of successive fractional separations may be increased to twelve, sixteen and even twenty-four.

By means of this invention inferior grades of diatomaceous earth may be treated for the production of extremely efficient materials. For example, a diatomaceous earth of rather inferior grade was dried and milled and the milled material then sent through six successive fractional air separators. The original material supplied to the system when tested for filtration efficiency on sugar liquor, in accordance with a standard testing method, indicated a flow rate of only 71% as compared with a standard commercial material. The residual material collected in and discharged by the sixth cyclone had a flow rate of 135% in comparison with the standard material, and constituted 63.5% of the total weight of material originally sent into the system. The extremely fine material which was removed by the fractional separation constituted 32.8% of the original material and was of extreme fineness, namely, the size of the particles did not exceed about 2 microns, thus rendering this particular material of great utility as a fine filler and polishing agent.

By a more extensive fractional classification, the original diatomaceous earth (having a flow rate of only 71%) was fractionated into three components consisting of 30% of the original material as a product having a filtration efficiency of 306% of the standard, 10% of a product having a filtration efficiency of over 100% and 50% of an extremely fine material adapted for use as a paint filler, rubber filler, etc.

In other words, the method of this invention makes it possible to utilize inferior grades of diatomaceous earth which would normally not be employed for use as a filter aid, and makes it possible to produce products from such inferior diatomaceous earths which are three times as effective as commercial filter aids. In addition, products may be prepared which are particularly adapted for use as fillers, and in which the particle size is below that which could be obtained by merely milling thoroughly a diatomaceous earth.

The following table gives the operating data on a separation made on a milled diatomaceous earth having a flow rate of 97% and a wet density of 16 lbs., per cubic foot. The wet density is obtained by suspending a quantity of the material in water and centrifuging these suspensions so as to compact the material in the bottom of the tube. From the volume occupied by the sample, the weight of the material per cubic foot may be computed and it thus represents substantially the maximum density to which the material may be compacted when in the presence of water and pressure. Materials of low wet density are normally more highly efficient filter aids than products which have a high wet density.

The original feed material having a flow rate of 97% was fractionally classified and the following table gives the characteristics of the residual material and of the fines removed every second pass until a total of fourteen passes or fourteen fractional removals were made.

	Suspended product				Fines removed				
No. Pass	Flow Rate	Wet D.	Percent Loss	Percent Yield	Flow R.	Wet D.	Percent Yield	Mesh 200	Mesh 325
2	122%	15.6	5.3	75.9	43	20.8	18.8	Tr.	Tr.
4	217%	13.9	7.7	61.0	60	19.5	31.3	Tr.	Tr.
6	286%	13.3	11.3	45.8	91	16.4	42.5	Tr.	Tr.
8	377%	13.3	10.2	38.3	122	14.9	51.5	1.7	4.0
10	451%	13.3	9.1	33.4	143	14.5	57.5	0.5	2.7
12	497%	13.0	9.4	29.0	181	13.0	61.6	1.0	3.2
14	584%	12.8	10.4	24.6	223	13.3	65.0	0.9	4.4

It is to be noticed that after 4 passes 61% was obtained as a residual product, such residual product having a flow rate of 217%. 31.3% of the material was removed in the form of a baghouse grade of material which was so fine that only a trace remained on 325 mesh. The wet density of this removed material was 19.5 lbs., per cubic foot, whereas the residual material had a wet density of only 13.9 lbs., per cubic foot. The loss in the system amounted to 7.7%, but such loss was in this instance merely due to the removal of large quantities of material for sampling purposes.

After 10 passes the original material had been divided into three components. Substantially 32% of the material had been removed in the form of a filter aid having an efficiency of about 100%; 33% of the material had a filtration efficiency of 451%, and approximately 35% of the material was in the form of an extremely fine filler.

After 14 passes the original feed had been fractionally classified into four component groups in the following proportions; approximately 35% was in the form of a fine filler, approximately 33% in the form of a filter aid having 100% efficiency, about 8% of the material was in the form of a filter air having an efficiency of about 200%, and 25% of the original material was in the form of a product having an efficiency of over 500%.

The original crude material fed into the system and discussed hereinabove was substantially a commercial material having an efficiency originally of 97% of a standard commercial product. It is thus seen that standard commercial filter aids may be selectively classified for the production of products having extraordinary properties. Furthermore, the process of fractional classification is not limited to crude or fresh milled diatomaceous products, but is also applicable to the fractional classification of calcined products.

Again, as stated hereinabove, the invention is not limited to the treatment of diatomaceous products, but may also be applied to fractional classification of all finely divided mineral materials containing components of different particle size, such components being individually more valuable than the heterogeneous mixture present in the original material.

Although in the drawing appended hereto and in the description of said drawing repeated reference has been made to the cyclonic type of air separating equipment, it is to be understood that the invention is not limited thereto. Any suitable form of separating equipment may be employed, such as for example, cyclones, stocking bag collectors or large settling chambers. Furthermore, it is not necessary in making these fractional classifications to employ a continuous chain of separate zones, but instead a smaller number of separating zones may be employed and the residual material repeatedly passed through the same cyclone or separating zone for further fractional classification.

What I claim is:

1. A method of classifying diatomaceous earth to produce fractional products consisting of particles of relatively homogeneous particle sizes, which comprises disintegrating coherent masses of diatomaceous material into large, intermediate, and small particles and (1) dispersing and suspending the finely divided disintegrated material containing the various sized particles in an aeriform current, (2) introducing the aeriform dispersion into a separating zone distinct from the dispersing zone, (3) separating in said separating zone small particles present in said dispersion together with the aeriform current from the residual dispersed material constituting the major proportion of the dispersed material, (4) passing the separated aeriform current bearing the small particles from said first mentioned separating zone into a second separating zone to separate and collect these particles from the aeriform current, and thereafter repeatedly subjecting the finely divided residual product constituting the major proportion of the dispersed material produced by step (3) to the sequence of steps (1) to (4) inclusive until the desired number of fractional products are produced.

2. A method of classifying diatomaceous earth to produce fractional products consisting of particles of relatively homogeneous particle sizes which comprises disintegrating coherent masses of diatomaceous material into large, intermediate and small particles approximating diatomic sizes and (1) mildly milling and suspending the finely divided disintegrated material containing the various sized particles in an aeriform current, (2) introducing the aeriform dispersion into a separating zone distinct from the dispersing zone, (3) separating in said separating zone small particles contained in said dispersion together with the aeriform current from the residual dispersed material constituting the major proportion of the dispersed material, (4) passing the separated aeriform current bearing the small particles from said first mentioned separating zone into a second separating zone to separate and collect these particles from the aeriform current, and thereafter repeatedly subjecting the residual product containing particles of various sizes constituting the major proportion of the dispersed material produced by step (3) to the sequence of steps (1) to (4) inclusive until the desired degree of classification is effected.

3. A method of classifying diatomaceous earth to produce fractional products consisting of particles of relatively homogeneous particle sizes which comprises disintegrating coherent masses of diatomaceous material into large, intermediate, and small particles and (1) dispersing and suspending the finely divided disintegrated material containing the various sized particles in an aeriform current, (2) introducing the aeriform dispersion into a separating zone distinct from the dispersing zone, (3) separating in said separating zone small particles contained in said dispersion together with the aeriform current from the residual dispersed material, constituting the major proportion of the dispersed material, (4) passing the separated aeriform current bearing the small particles from said first mentioned separating zone into a second separating zone to separate and collect these particles from the current, and thereafter repeatedly subjecting the residual product containing particles of various sizes constituting the major proportion of the dispersed material produced by step (3) to the sequence of steps (1) to (4) inclusive until the desired degree of classification is effected, and combining a plurality of the fractions produced by the repetition of step (4).

4. A method of classifying diatomaceous earth to produce fractional products consisting of particles of relatively homogeneous particle sizes, which comprises disintegrating coherent masses of diatomaceous material into large, intermediate, and small particles and (1) dispersing and suspending the finely divided disintegrated material containing the various sized particles in an aeriform current, (2) introducing the aeriform dispersion into a separating zone distinct from the dispersing zone (3) separating in said separating zone small particles present in said dispersion together with the aeriform current from the residual dispersed material constituting the major proportion of the dispersed material, (4) passing the separated aeriform current bearing the small particles from said first mentioned separating zone into a second separating zone to separate and collect these particles from the aeriform current, and thereafter repeatedly subjecting the finely divided residual product constituting the major proportion of the dispersed material produced by step (3) through at least five more cycles to the sequence of steps (1) to (4) inclusive until the desired number of fractional products are produced.

5. A method of classifying diatomaceous earth to produce fractional products consisting of particles of relatively homogeneous particle sizes, which comprises disintegrating coherent masses of diatomaceous material into large, intermediate, and small particles, drying the earth to a moisture content not in excess of 10% and (1) dispersing and suspending the finely divided disintegrated material containing the various sized particles in an aeriform current, (2) introducing the aeriform dispersion into a separating zone distinct from the dispersing zone, (3) separating in said separating zone small particles present in said dispersion together with the aeriform current from the residual dispersed material constituting the major proportion of the dispersed material, (4) passing the separated aeriform current bearing the small particles from said first mentioned separating zone into a second separating zone to separate and collect these particles from the aeriform current, and thereafter repeatedly subjecting the finely divided residual product constituting the major proportion of the dispersed material produced by step (3) to the sequence of steps (1) to (4) inclusive until the desired number of fractional products are produced.

6. A method of classifying diatomaceous earth to produce fractional products consisting of particles of relatively homogeneous particle sizes, which comprises disintegrating coherent masses of diatomaceous material into large, interme-

diate, and small particles and (1) dispersing and suspending the finely divided disintegrated material containing the various sized particles in an aeriform current, (2) introducing the aeriform dispersion into a cyclonic separator constituting a separating zone distinct from the dispersing zone, (3) separating in said separating zone small particles present in said dispersion together with the aeriform current from the residual dispersed material constituting the major proportion of the dispersed material, (4) passing the separated aeriform current bearing the small particles from said first mentioned separating zone into a second cyclonic separator constituting a second separating zone to separate and collect these particles from the aeriform current, and thereafter repeatedly subjecting the finely divided residual product constituting the major proportion of the dispersed material produced by step (3) to the sequence of steps (1) to (4) inclusive until the desired number of fractional products are produced.

7. A method of classifying diatomaceous earth to produce fractional products consisting of particles of relatively homogeneous particle sizes which comprises disintegrating coherent masses of diatomaceous material into large, intermediate and small particles approximating diatomic sizes and (1) mildly milling and suspending the finely divided disintegrated material containing the various sized particles in an aeriform current by passage through a centrifugal fan constituting a milling and dispersing zone, (2) introducing the aeriform dispersion into a separating zone distinct from the dispersing zone, (3) separating in said separating zone small particles contained in said dispersion together with the aeriform current from the residual dispersed material constituting the major proportion of the dispersed material, (4) passing the separated aeriform current bearing the small particles from said first mentioned separating zone into a second separating zone to separate and collect these particles from the aeriform current, and thereafter repeatedly subjecting the residual product containing particles of various sizes constituting the major proportion of the dispersed material produced by step (3) to the sequence of steps (1) to (4) inclusive until the desired degree of classification is effected.

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