

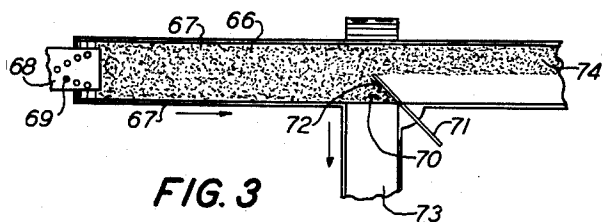
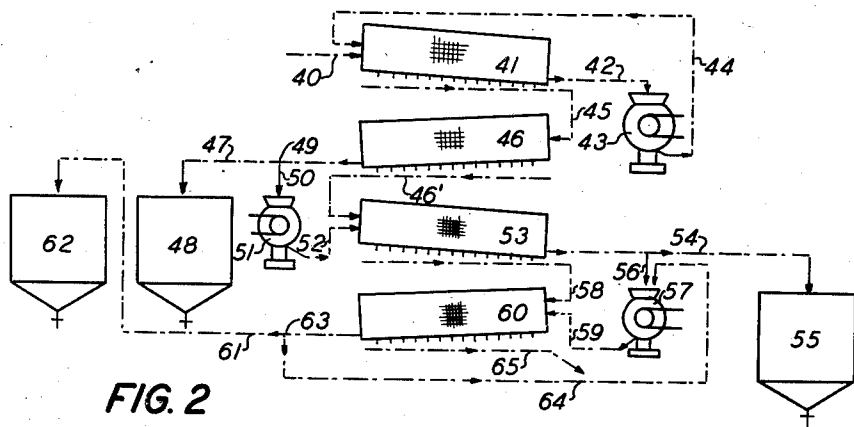
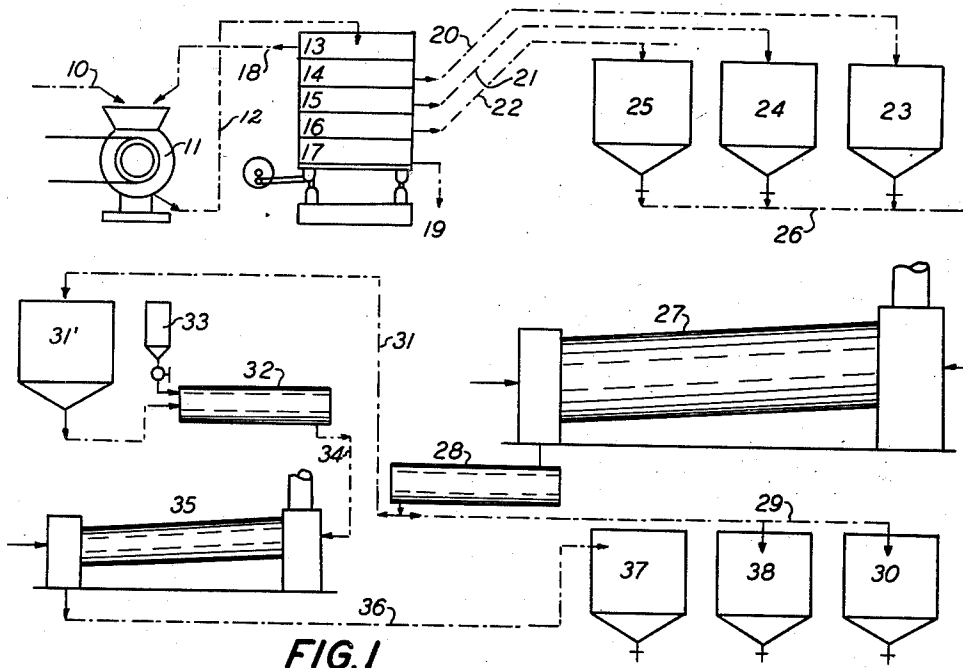
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CALCINED DIATOMACEOUS EARTH PRODUCT

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CALCINED DIATOMACEOUS EARTH  
PRODUCTCharles A. Frankenhoff, Scarsdale, N. Y., assignor  
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This invention relates to the heat treatment of crude diatomaceous earth and to the segregation of the earth, before or after such treatment, into products of different size ranges having varied utilities.

A purpose of the invention is to obtain certain commercial products, to wit: a bedding material for fowl housing; an air and gas dehumidifying agent, and an agent for cleaning floors, all of these uses requiring a highly developed liquid-absorbing characteristic, the greatest possible resistance to crushing and abrasion and complete freedom from tendency toward mudding with water.

A purpose of the invention is to obtain the above named products by a systematic segregation and heat treatment of the crude diatomaceous earth, the segregation being so controlled as to utilize advantageously the whole of the earth as mined.

The invention is capable of two modifications, in the first of which the crude lump earth is crushed, is then segregated into fractions of desired particle size ranges, and the fractions are then separately heat-treated. In the second alternative the heat treatment is applied to the crude lump earth, which is thereafter crushed and segregated into fractions.

The first alternative will be described with reference to the attached drawings, in which

Fig. 1 is a diagram and flow sheet of the process and of an assemblage of apparatus units suitable to putting it into effect;

Fig. 2 is a diagram of a modified crushing and screening operation, and

Fig. 3 is a detail of the essential parts of a device for controllably dividing a stream of solid fragments into two streams.

In Figs. 1 and 2, elevating and conveying elements are not illustrated but are indicated by broken lines and the directions of movement by arrowheads.

The crude diatomaceous earth to be used for the present purpose should be selected carefully. It is desirable that it should be moderately hard in the crude state and it should have a Gardner-Coleman water-absorption factor not much below 80%. (Gardner, Paints, Varnishes, etc., 7 ed., p. 541.) The content of clay should be as low as possible. A fresh water earth is preferable to one of marine origin as being less likely to be laminated and as tending to crush to cubical rather than to plate-like fragments. The variety of earth mined near Basalt in Montgomery Pass, Mineral County, Nevada is a typically desirable earth for this purpose, though the invention is by

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no means limited to the use of this particular raw material.

If the crude earth be wet, as is often the case, it should be given a preliminary drying to render it brittle enough to crush freely. Ordinarily the water content should be reduced to 20% or less, by air drying or by heating at a low temperature. The run-of-mine crude, which usually occurs in relatively large lumps, is then fed into the plant as by a conveyor 10 and passed through a crusher 11. This element may be of any type adapted to crushing rock, preference being given to rolls or to breakers of the jaw or gyratory type over forms, such as the disc breaker, which tend to the production of undesired fines. As will later be seen, the object is to produce by crushing a relatively even gradation of size from a maximum of  $1\frac{1}{4}$ " or 1" to a minimum of about 30 U. S. standard mesh, with as little finer product as possible.

The crushed raw earth is passed by an elevator 12 to a succession of screens 13, 14, 15, 16 with a final collecting tray 17. These are illustrated in Fig. 1 as a stack of shaking or vibrating screens, but the unit may consist of a series of sloping screens or of a four-stage trommel. The crushed material, if not unduly burdened with fine powder, screens readily.

The following table gives the preferred openings for these screens, in terms of square mesh woven wire. There is, however, a reasonable degree of latitude as regards these sizes and the tolerance stated are either plus or minus. For convenience, the qualification "about" will be used to include the tolerance, thus "about 1" will be understood to mean  $1\frac{1}{4}" \pm \frac{1}{4}"$ , or any size within the range of  $1\frac{1}{4}"$  to  $\frac{3}{4}"$ .

|                                                                    |
|--------------------------------------------------------------------|
| Screen 13—preferred mesh 1", tolerance $\frac{1}{4}"$              |
| Screen 14—preferred mesh $\frac{1}{2}"$ , tolerance $\frac{1}{8}"$ |
| Screen 15—preferred mesh $\frac{3}{8}"$ , tolerance $\frac{1}{8}"$ |
| Screen 16—preferred mesh .033", tolerance .010"                    |

These screens will deliver five fractions, as follows:

Fraction A—over screen 13—particle size about 1" ( $1\frac{1}{4}"$  to  $\frac{3}{4}"$ ) and coarser—returned to the crusher by conveyor 18 to be again reduced in size;

Fraction B—through screen 13 and over screen 14—particle size range from about 1" ( $1\frac{1}{4}"$  to  $\frac{3}{4}"$ ) to about  $\frac{1}{2}"$  ( $\frac{5}{8}"$  to  $\frac{3}{8}"$ );

Fraction C—through screen 14 and over screen 15—particle size range from about  $\frac{1}{2}"$  ( $\frac{5}{8}"$  to  $\frac{3}{8}"$ ) to about  $\frac{3}{8}"$  ( $\frac{1}{4}"$  to  $\frac{1}{8}"$ );

Fraction D—through screen 15 and over screen

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16—particle size range from about  $\frac{1}{8}$ " ( $\frac{1}{4}$ " to  $\frac{1}{8}$ "') to about .033" (.043" to .023"');

Fraction E—through screen 16—maximum particle size about .033" (.043" to .023"). This fraction is discharged from the system at 19 and may be used for making filter aids, filling and bodying materials or other diatomaceous earth products.

Some crude earths, when subjected to a given crushing operation, yield a smaller proportion of finely divided particles than other earths similarly treated. If the proportion of fines produced by the crushing step be sufficiently low, fractions D and E may be combined by omitting screen 16. In this case fraction D will have a maximum particle size of about  $\frac{1}{8}$ " ( $\frac{1}{4}$ " to  $\frac{1}{8}$ "') and will include all finer particles. The proportion of particles passing a screen of .033 mesh should not exceed about 25% of the weight of the fraction.

Fractions B, C and D, as they come from the screening unit, are delivered by elevators 20, 21 and 22 into bins 23, 24 and 25 in which they are accumulated for further separate treatments.

When a sufficient quantity of fraction B has been collected in bin 23, it is withdrawn into a conveyor 26 by which it is introduced in a stream into an internally fired, rotary kiln 27, such, for example, as illustrated in U. S. Patent 1,727,036 to Rank and Frease, or of the type ordinarily used for clinkering cement. A screw conveyor is best adapted to handling the screen raw material, but care should be taken that this element be of ample capacity, as the screened raw lumps are rather fragile and will break down if crowded.

The kiln, of whatever pattern, is somewhat strongly fired, preferably at 1500° to 2000° Fahr. The kiln discharges into a rotary or other cooler 28, in which the calcined product is brought down to a temperature at which it may be passed by a conveyor 29 into a storage bin 30. The calcined product of the heat treatment of fraction B is particularly adapted for use in poultry yards and houses, as will be described.

In the heat treatment of fraction C the kiln is preferably operated at a somewhat lower temperature, for example within the range 1300° to 2000° Fahr. After passing from bin 24 through kiln 27 and cooler 28 the calcined fraction is delivered by an elevator 31 to a bin 31' from which it flows to a mixer 32. In this element, which is preferably of the rotating barrel type, the calcined earth is mixed with a saturated aqueous solution of a highly hygroscopic salt, as for example calcium chloride, zinc chloride or phosphorus pentachlorid, flowing from a tank 33.

The quantity of solution introduced at this point should be the maximum which can be absorbed completely by the earth, leaving it saturated but not externally wet. The salt saturated earth passes continuously from the mixer over a conveyor 34 to a dryer indicated at 35, which may desirably be the same or another internally fired, rotary kiln. If separate units are preferred, the drying kiln may be smaller and of lighter construction than kiln 27. In the dryer the saturated earth is contacted with fire gases at a relatively low temperature (e. g., 400° to 600° Fahr.) until all the water of the added solution is driven off. From the dryer the substantially anhydrous product passes over conveyor 36 to a storage tank 37, which should be closed to prevent loss of activity by contact with moist air. This product is adapted to the dehumidification of air and other gases, as will be described.

In the heat treatment of fraction D the material collected in bin 25 is transferred by conveyor

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26 to kiln 27 in which it is calcined, preferably within the temperature range 1300° to 1800° Fahr. The calcined product passes from cooler 28 by conveyor 29 to a bin 38. This product is adapted to the cleansing of oily floors, as will be described.

The calcination temperature ranges above recited are preferred but are not critical. The essential is to bring the temperature to the level at which the water of hydration of the silica is completely driven off. As will be shown, the increased water absorption produced by calcination at a relatively low temperature may be lost on further temperature elevation, while the important characteristics of hardness and resistance to abrasion improve as the temperature rises. The optimum temperature in any given case will be found by balancing the importance of absorption and toughness for the specific use to which the product is to be put.

The alternative method of operation first referred to differs from that above described in passing the crude earth, in run-of-mine form or after coarse crushing, through the kiln, the calcined lumps being then crushed and segregated by screening as above described. As in this operation all of the final products have been treated at the same temperature, it is desirable to calcine at a moderate temperature level, as for example from 1300° to 1800° Fahr.

The quality of the final products is substantially the same in either case, and the advantage of a slight simplification of plant in the use of the alternative may be offset against the loss of ability to vary the calcining temperatures of the different products and by the somewhat lesser value of fines produced in crushing and screening the precalcined earth.

A highly desirable feature of either modification of the process above described lies in the utilization of substantially the whole of any given supply of diatomaceous earth, by subdividing it into a series of products each of which has the size range most suitable for the particular use to which it is put.

The use of a succession of screens avoids any overlap of size ranges or gap between them, but as the relative demands for different products will vary from time to time it is highly important to provide some form of control over these quantitative relations. This is particularly important if the alternative method (of precalcination) be adopted, as while any excess quantity of crude earth of any size range, obtained in increasing the yield of another fraction, can be utilized by reducing it to powder, the utility of calcined earth in powdered form is somewhat limited.

As the screening system can do no more than to segregate into suitable size ranges the mixture of fragments coming to it, control over the relative yield of each fraction must go back in some manner to the manipulation of the crushing step. A very rough and imperfect control may be effected by merely varying the spacing of the jaws or rolls of the crusher but in most cases this control will be found wholly inadequate.

Complete control over the relative quantities of different size ranges may be effected by the manipulation shown in the form of a flow sheet in Fig. 2. Referring to this figure, the run-of-mine earth (either crude or precalcined) enters the system at 40 and passes through a trommel or other screening element 41 which passes all particles below the maximum size range of frac-

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tion B: e. g., this screen may be of  $1\frac{1}{4}$ " to  $\frac{3}{4}$ " mesh.

The oversize from this screen passes over conveyor 42 to a primary crusher 43 of relatively large capacity. This element should be of a type adapted to handling up to large lumps without subjecting them to a grinding action. A toothed roll crusher is preferred, the spacing of the lowermost and closest rolls being capable of adjustment over a considerable range, as for example from 1" to 2". The roll spacing in the primary crusher will require variation with changes in the friability of the lump earth, such as may be produced by variation in water content.

The product of the primary crusher, which will have a wide size range, is returned by conveyor 44 to the roughing screen 41, from which the oversize returns to the crusher together with the oversize from the lump feed. By this cycling, the entire feed will ultimately be passed through the roughing screen, even with a jaw or roll spacing much greater than the screen mesh. This procedure is advantageous over crushing to the mesh of the roughing screen in a single pass, in reducing the yield of fines and in facilitating control over the relative proportions of different fractions.

The screenings from trommel 41 pass as at 45 into a second screen or trommel 46 which may, for example, be of  $\frac{5}{8}$ " to  $\frac{3}{8}$ " mesh. The oversize retained by this screen, which will correspond with the above described fraction B, may be in quantity either above or below the required proportion of the total feed. This quantity is subject to control in the following manner.

If the yield of this fraction is deficient, the spacing of the bottom rolls of crusher 43 is widened to increase the yield of larger size fragments, this widening being continued until the desired proportion of the raw feed is retained by screen 46. This fraction is transferred by conveyor 47 to a storage bin 48.

If the yield of fraction B is excessive, the stream of oversize passing from this screen is divided at point 49 (in a manner suggested hereinafter with reference to Fig. 3), a proportion equal to the requirement for this fraction passing on to bin 48, the excess being diverted onto a conveyor 50 by which it is passed to a secondary crusher 51. This element may be of much smaller capacity than the primary crusher and should have a roll adjustment ranging upwardly from the mesh dimension of the third screen 53.

The undersize from screen 46 passes as at 46' to a third screen or trommel 53 which may, for example, be of  $\frac{3}{8}$ " mesh. This element also receives the crushed product from secondary crusher 51 by way of conveyor 52.

The retention on this screen will have a particle size range corresponding with fraction C above described, and again may be deficient or excessive in quantity. The yield of this fraction, if deficient, is adjusted by widening the roll spacing of crusher 51 and, if that be insufficient for the purpose, of primary crusher 43 also. If the yield be excessive, it is divided in the manner above described, the required proportion passing as at 54 into a bin 55 while the excess is diverted as at 56 into a third crusher or other comminuting device 57. This element, if a crusher, should have a roll spacing adjustment varying upwardly from the mesh dimension of the final screen 60, and may be of small capacity.

The screenings from the third screen 53 pass

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as at 58 into the final screen 60 which may, for example, be of #30 mesh. This screen also receives the crushed product from element 57 by way of conveyor 59.

The retention on screen 60, which will have a particle size range corresponding to fraction D above described, passes by conveyor 61 to a bin 62. As before, any excess yield is diverted at 63 onto a conveyor 64 by which it is returned to third crusher 57. The fines passing through screen 60 are rejected from the system as at 65. A deficiency in yield of this fraction is overcome by widening the adjustment of one or more of the preceding crushers.

The manipulation just described is highly desirable in instances in which the fines are not adapted to the making of other diatomaceous earth products, or where facilities for that purpose are lacking. The yield of fines is reduced to the minimum by excluding from the crushers any material finer than that which the individual unit is to produce. The effect of an intermixture of undersize fragments, and particularly of fine particles, in promoting the production of further quantities of fines, is well known.

The two secondary crushers 51 and 57 may be dispensed with and the diverting conveyors arranged to return excess quantities to primary crusher 43. This change will produce at least a moderate increase in the yield of fines and is undesirable unless there is a satisfactory outlet for this fraction.

In either modification, the system described offers a means for exact control of the quantitative relations of the three desired products. From the operative point of view such control is of major importance, particularly as the harder earths which are best suited for the manufacture of these products are not, as a rule, well adapted to the manufacture of filter aids.

A preferred means for dividing a stream of solid particles, such as the products retained by screens 46, 53 and 60, is illustrated in Fig. 3. In this figure, 66 represents a flat conveyor belt receiving the stream of solid to be divided, this belt running between stationary side walls 67 to prevent spillage. The stream of solids flows onto the belt from a spout 68 so formed as to distribute the stream as equally as possible over the width of the belt. A plurality of upstanding pegs 69 will aid distribution if the spout be steeply inclined. The conveyor, of which only a fragment is shown, leads to any place of reception of a portion of the stream, as for example one of the bins above referred to.

At any convenient point along the length of this belt, one of the side walls 67 is interrupted to leave a gate 70. At this point a scraper 71, riding on the belt at an acute angle, is so mounted as to swivel or to be movable endwise to cover a variable portion of the width of the belt. At whatever position this scraper is placed, the portion 72 of the main stream encountering the face of the scraper is diverted onto a lower belt 73 by which it is conveyed to another destination, as for example one of the secondary crushers supra, the remaining proportion 74 proceeding onward to the bin. By moving the tip of this scraper in or out, any desired proportion of the stream of solid particles may be directed to either destination.

#### REQUIRED CHARACTERISTICS OF PRODUCTS

As above said, the products of the described operation are: a coarse fraction used as fowl bedding material; a fraction of intermediate

particle size carrying a hygroscopic salt and used as a dehumidifying agent, and a relatively finely divided fraction used as a floor cleansing agent.

These three products must have a full development of the same characteristics in order to be of the best quality. The ability to absorb liquids must be as great as possible; the products must have a satisfactory load-bearing characteristic; they must resist abrasion to the maximum extent, and they must be free from any tendency to slack or mud when wetted with water or aqueous liquids.

Diatomaceous earth, either raw or calcined, is an excellent absorbent for liquids, and this property of the crude earth may be but is not necessarily improved by calcination. In respect to the other three requirements the raw earth is highly defective, to such extent as to render it of little if any utility for any one of these three uses, and the effect of calcination is uniformly to improve these properties and thereby to render the products not merely useful but of highly superior quality.

The effects of calcination on each of the four desired properties of the material is illustrated by the results of the experiments described below.

#### Experiment 1.—Effect on absorption factor

A sample of raw Nevada earth was dried at low temperature to remove only free water and other samples of the same earth were calcined at different temperatures. The Gardner-Coleman absorption factor was then determined on each sample, using water as the vehicle, with the results following:

|                                                | Per cent weight |
|------------------------------------------------|-----------------|
| Earth dried only, water absorption.....        | 95              |
| Earth calcined at 1200° F., water absorption.. | 114             |
| Earth calcined at 1500° F., water absorption.. | 100             |
| Earth calcined at 1800° F., water absorption.. | 93              |

In this particular case the gain in absorption produced at 1200° was lost by continuing heating to 1800°. These exact figures might not obtain with other earth, but the general rule holds that absorption is improved up to a certain optimum temperature and thereafter declines, while the other desired characteristics continue to improve as the temperature rises.

#### Experiment 2.—Effect on crushing strength

Rectangular slips measuring 20 x 17 x 15 mm. were cut from large lumps of a raw Nevada earth and of the same earth after calcining at 1500° F. The slips were then crushed hydraulically through their shorter dimension and the break-down pressure calculated to pounds per square inch, with the following results:

|                                                           |    |
|-----------------------------------------------------------|----|
| Raw earth broke down at 400/410 pounds per square inch    | 60 |
| Calcined earth broke down at 2300/2350 pounds per sq. in. |    |

#### Experiment 3.—Effect on abrasion resistance

Samples of a raw Nevada earth and of the same earth after calcining at 1500° Fahr. were sized to pass a screen of 0.380" square opening and to be retained on a screen of 0.295" opening. A weighed quantity of each sample was then placed in a small cubical box containing 1" cubes of hard wood, the box was rotated for 15 minutes, and the fragments passing a 6 mesh screen were weighed as abrasion loss, with the following results:

Per cent weight

|                                     |    |
|-------------------------------------|----|
| Abrasion loss, raw sample.....      | 18 |
| Abrasion loss, calcined sample..... | 6  |

#### Experiment 4.—Effect on resistance to mudding

Samples of raw and of calcined earth similar to those used in the third experiment were placed in a laboratory ball mill and covered with water, the usual grinding balls being omitted. The mill was then rotated at 42 R. P. M. for 5 minutes, after which any resultant fines were washed through a 6 mesh screen, dried and weighed as mudding loss, with the following results:

|                                    | Per cent weight |
|------------------------------------|-----------------|
| Mudding loss, raw sample.....      | 13.6            |
| Mudding loss, calcined sample..... | .0              |

The fowl bedding material above referred to—the coarser segregated fraction of calcined diatomaceous earth—has properties which distinguish it clearly from other materials which have been used for this purpose.

It is obviously and clearly distinguished from such organic materials as straw and the like by being wholly mineral and not subject to fermentation or decay. Raw diatomaceous earth has heretofore been used for covering the floors in poultry houses and its high absorptive power would render it highly suitable for such use were not its other properties such as to render it unsuitable. The fragility of the crude earth lumps and their tendency to slack and mud when wetted limit the use of this material to protected situations, and when excluded from contact with rain or ground water the lumps of crude earth are rapidly converted into dust when walked over. Calcination of the earth cures these defects and yields a product which maintains its lump form when subjected to foot traffic and through repeated wettings and dryings, and which dries out rapidly after being wetted. These are major advantages which give this product a new and enhanced usefulness.

The particle size range recited for fraction B is preferred for this purpose, but is not critical. The ability of the calcined lumps to bear up under load and thus to maintain their original size permits the addition of considerable proportions of smaller particles. While it is undesirable to include lumps larger than about 1", there is no necessary lower particle size limit other than that the product should be substantially free from dust and particles smaller than about .025".

This product may be defined as calcined diatomaceous earth (i. e., diatomaceous earth free from water of hydration), in lumps and particles, preferably within the size range from about 1" to about  $\frac{3}{16}$ " and permissibly within the size range from about 1" to about .033", having a high resistance to crushing and abrasion and substantially free from tendency to slack when wetted with water.

The calcined diatomaceous earth fraction used as support for a hygroscopic salt in the making of a dehumidifying agent for air and gases has distinguishing properties which render it uniquely useful for this purpose.

Attempts have been made heretofore to take advantage of the high absorption factor of diatomaceous earth as the supporting element in a dehumidifying agent, but the tendency of the raw earth to slack and mud has limited these applications to the use of the earth in powdered form, either as such or in lumps produced by

the use of a binding agent such as Portland cement.

These powders, when used as such, have an absorbing value equal or superior to that of the same earth in lump form, but they cannot be formed into a gas-permeable bed and must be spread out in thin sheets of which only the exposed surface is effective. Further, the exhausted powder is difficult to regenerate by drying because of the tendency of the absorbed salt to cake or even to sinter when heated. The lumps or shapes formed with the use of binders have a much reduced absorption factor and are of little utility as dehumidifying agents.

The calcined granules, on the other hand, do not slack when wetted, have a strong resistance to abrasion and therefore may be regenerated and used repeatedly. They form freely permeable beds and have unimpaired crushing strength when wet, so that they may be used in drying columns of any desired height. In regenerating, they may be heated as rapidly and strongly as may be desired, short of a temperature at which the specific salt used begins to attack the earth, this being far above any temperature practically useful.

The absorption factor for strong salt solutions is somewhat greater than that for water on the weight basis, somewhat less when volumes are considered. This is illustrated by the results of the experiment following:

*Experiment 5.—Absorption of calcium chloride solution*

A raw diatomaceous earth, and the same earth after calcination at 1500° Fahr., were reduced to fragments of identical size ranges (over .295" and through .380" mesh) and the absorption factor determined, using both water and a 42% aqueous solution of calcium chloride, with the following results:

|                                     | Per cent weight |
|-------------------------------------|-----------------|
| Water absorption, raw earth         | 77              |
| Solution absorption, raw earth      | 81              |
| Water absorption, calcined earth    | 81              |
| Solution absorption, calcined earth | 89              |

The quantity of hygroscopic salt introduced into the earth may be varied according to the degree of desiccation to which it is necessary to bring the gas. In the case just recited, 1 pound earth, after saturation and drying, will retain 0.39 pound of the salt and the resultant 1.39 pounds of agent will reabsorb 0.52 pound water, or 0.37 pound per pound agent, before any leakage of liquid can take place. Under these conditions the gas will be in contact only with solid calcium chloride or its saturated solution and will be brought to the highest degree of desiccation of which calcium chloride is capable at the operating temperature.

If a smaller quantity, for example 0.1 pound of the salt be introduced into the earth, 1 pound of the resultant agent will absorb a larger quantity, about 0.65 pound of water before leakage begins, but under these conditions the gas will be in contact with solution of continuously diminishing strength and will be dried only to a higher residual moisture content.

The particle size range recited for fraction C is preferred for the manufacture of a dehumidifying agent, but is not critical. It is undesirable to go much above a maximum particle size of about  $\frac{1}{2}$ " because of the reduction of surface area in relation to volume incurred as particle

size increases. At the other end of the range, however, the sole requisite is to maintain a gas-contacting bed open and permeable. To this end it is desirable to exclude all extreme fines (dust) and substantially to exclude particles smaller than about .033".

It is preferable to make the product in the manner first described, by mixing the granular earth with a strong solution of the hygroscopic salt and driving out the water of solution, complete and even dissemination of the salt through the earth being attained in that manner and the risk of stratification avoided. Under some circumstances, however, and particularly where the proportion of salt is relatively small, it suffices to mix the solid salt, in granular or flake form, with the granular earth. Such physical admixture will have less desiccating ability initially than the impregnated earth, because of the reduced contact area, but in its first use the salt will be dissolved and the solution absorbed by the earth grains.

This product of the invention may be defined as calcined diatomaceous earth (i. e., diatomaceous earth free from water of hydration), in lumps and particles, preferably within the size range from about  $\frac{1}{2}$ " to about  $\frac{3}{8}$ " and permissibly within the size range from about  $\frac{1}{2}$ " to about .033", having a high crushing strength and resistance to abrasion and free from tendency to slack when wetted with aqueous solutions, and mixed or impregnated with a hygroscopic salt, preferably calcium chloride.

Fraction C is also particularly suitable as a support for liquid or liquefiable cracking catalysts such as the phosphoric acids, all of which fuse at relatively low temperatures. The natural earth is unsuitable for this purpose by reason of its low crushing strength and its tendency to slack and slough on the application of the acid. In the use of the calcined product, the high absorption factor permits a large proportion of the active agent to be retained, the crushing strength of the granules is such as to allow contact columns to be of any desired height, and the spent agent may be regenerated many times without degrading the particle size.

The calcined product of smallest size range (fraction D above described or its equivalent) has properties which render it highly valuable for cleansing the floors of garages, machine shops, oil refinery buildings and others which are subject to spillage of oils, greases and caustic liquids. These properties distinguish it clearly from materials such as sand, sawdust and granular clays heretofore used for that purpose.

Sand is low in absorptive power in relation to weight, and sawdust, which is much better in that respect, is difficult to dispose of after use. Carefully selected clays of the fuller's earth type are as highly absorbent as sawdust, but in that respect are decidedly inferior to diatomaceous earth. This is illustrated by the results of the following experiment:

*Experiment 6.—Relation of absorption to weight*

A granular clay or fuller's earth marketed and rather widely used for this purpose was subjected to the usual screen test to determine its particle size range, the result being:

|               | Per cent |
|---------------|----------|
| Above 10 mesh | 2.9      |
| 10 to 20 mesh | 55.5     |
| 20 to 30 mesh | 26.3     |

|                     | Per cent |
|---------------------|----------|
| 30 to 65 mesh ----- | 12.9     |
| Below 65 mesh ----- | 2.4      |

A mixture of granules of diatomaceous earth of the same screen analysis was then prepared and the following tests made on the two materials:

|                                           |      |
|-------------------------------------------|------|
| Water absorption, clay product            |      |
| per cent weight--                         | 49   |
| Water absorption, diatomaceous earth      |      |
| per cent weight--                         | 99   |
| Weight per cubic foot, clay product       |      |
| pounds--                                  | 39.6 |
| Weight per cubic foot, diatomaceous earth |      |
| pounds--                                  | 23.3 |

From which it follows that one pound or other unit weight of the calcined diatomaceous earth will absorb about twice the quantity of liquid taken up by the clay product and, size ranges being equal, will cover about double the area when spread in layers of equal thickness.

A material advantage attending the use of calcined diatomaceous earth for floor cleansing is that it does not track. The absorption of oil or other liquid from a floor, particularly if the latter is of cement, requires at least a short and sometimes an extended period. During this period it will usually be walked over. The clays and fuller's earths heretofore used for this purpose, and to an even greater extent crude diatomaceous earth, grind down to an impalpable powder under foot traffic, and in a short time this powder may be tracked over other floors not undergoing treatment, requiring a further cleaning operation.

In the use of calcined diatomaceous earth, the larger fragments of which may fracture when stepped on but which will not grind down to a fine powder unless spread very thinly over a hard surface, tracking is practically absent and particular floor areas may be left covered with the absorbent granules for as long as may be needed, without fouling adjacent surfaces.

The particle size range specified above for fraction D, from about  $\frac{1}{16}$ " to about .033", is preferred as giving the best balance of fine and coarse particles for maximum absorption and least abrasion. It is not desirable to include any material proportion of particles larger than about  $\frac{1}{16}$ " as the larger granules make poorer contact with the oil soaked surface and remove the oil more slowly. At the other end of the range, a limited proportion, up to about 25%, of particles finer than about .033" may be included, these fine particles tending to accelerate absorption and being protected from tracking by admixture with the larger granules.

This product of the invention may be defined as calcined diatomaceous earth (i. e., diatomaceous earth free from water of hydration), in granules preferably ranging in size from about  $\frac{1}{16}$ " to about .033" but permissibly including not more

than 25% of particles smaller than about .033", the earth composing said granules having a high crushing strength and resistance to abrasion.

This application is a division of my copending application Serial No. 665,986, filed April 30, 1946 under the title "Calcined diatomaceous earth products."

I claim as my invention:

1. An anhydrous, freely permeable, granular dehumidifying agent for air and other gases, consisting essentially of massive particles of diatomaceous earth of about 0.5 inch to about 0.033 inch size prepared by calcining a raw diatomaceous earth capable of absorbing not less than about 80% of water by weight, at a temperature of about 1200° F. to about 1800° F., said particles having a water absorption of about 93 to about 114% by weight, an abrasion loss not exceeding about 6% by weight, being free of water of hydration and from the tendency to slack when wetted by water, and having a high crushing strength, said particles containing a hygroscopic salt in proportions of not less than 0.1 lb. nor more than 0.39 lb. per pound of said calcined particles.
2. The composition of claim 1 wherein the hygroscopic salt is calcium chloride.
3. An anhydrous freely permeable granular dehumidifying agent for air and other gases, consisting essentially of massive particles of diatomaceous earth of about 0.5 inch to about 0.033 inch size prepared by calcining a raw diatomaceous earth capable of absorbing not less than 80% of water by weight, at a temperature of about 1200° F. to about 1800° F., said particles having a water absorption of about 81% to about 114% by weight, an abrasion loss not exceeding about 6% by weight, being free of water of hydration and from the tendency to slack when weighted by water, and having a high crushing strength, said particles containing a hygroscopic salt in proportions of not less than 0.1 lb. nor more than 0.39 lb. per pound of said calcined particles.

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