

May 9, 1961

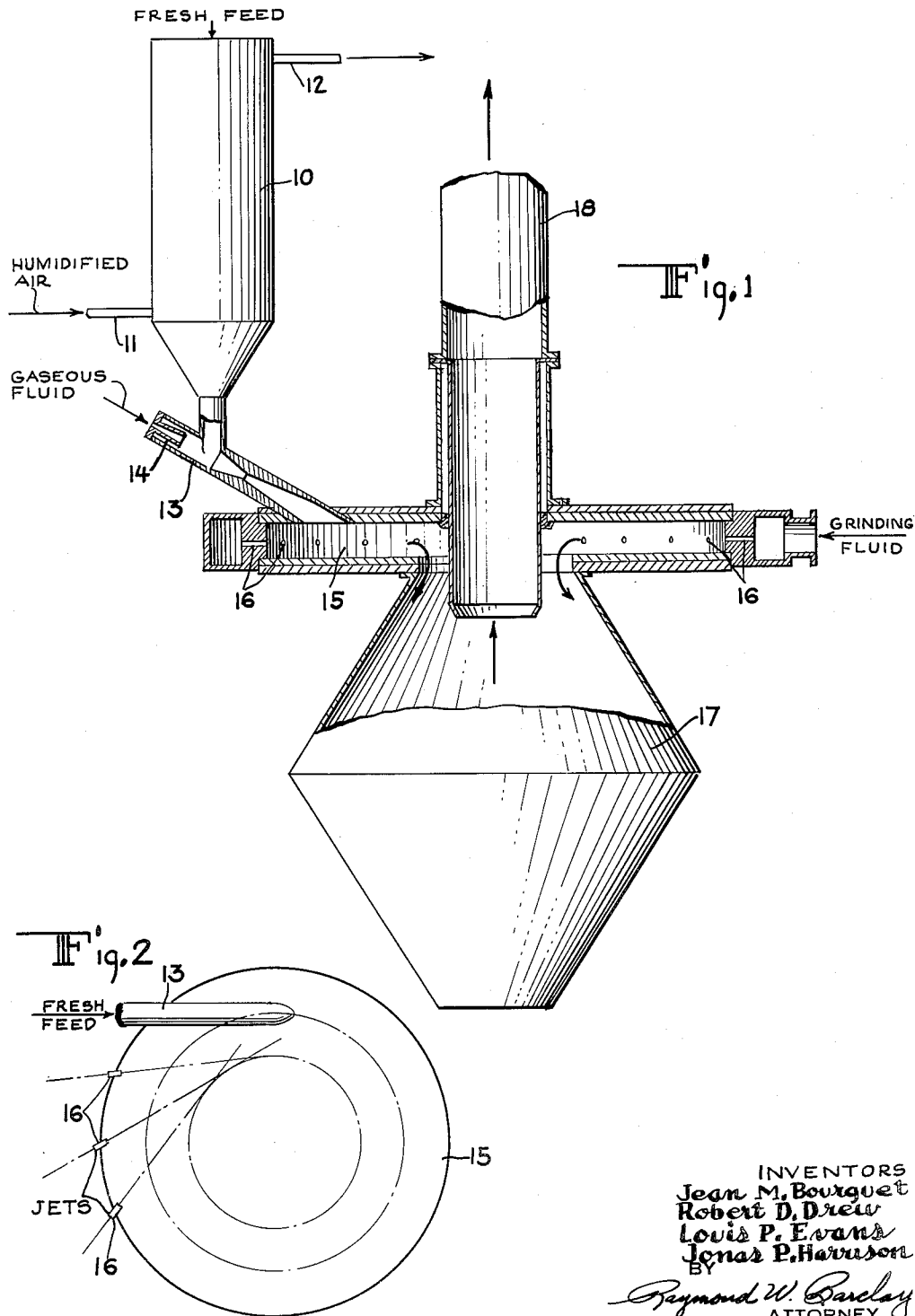
J. M. BOURGUET ET AL

2,983,453

METHOD OF PULVERIZATION

Filed March 13, 1958

2 Sheets-Sheet 1



INVENTORS
Jean M. Bourguet
Robert D. Drew
Louis P. Evans
Jonas P. Harrison

BY *Raymond W. Barclay*
ATTORNEY

May 9, 1961

J. M. BOURGUET ET AL

2,983,453

METHOD OF PULVERIZATION

Filed March 13, 1958

2 Sheets-Sheet 2

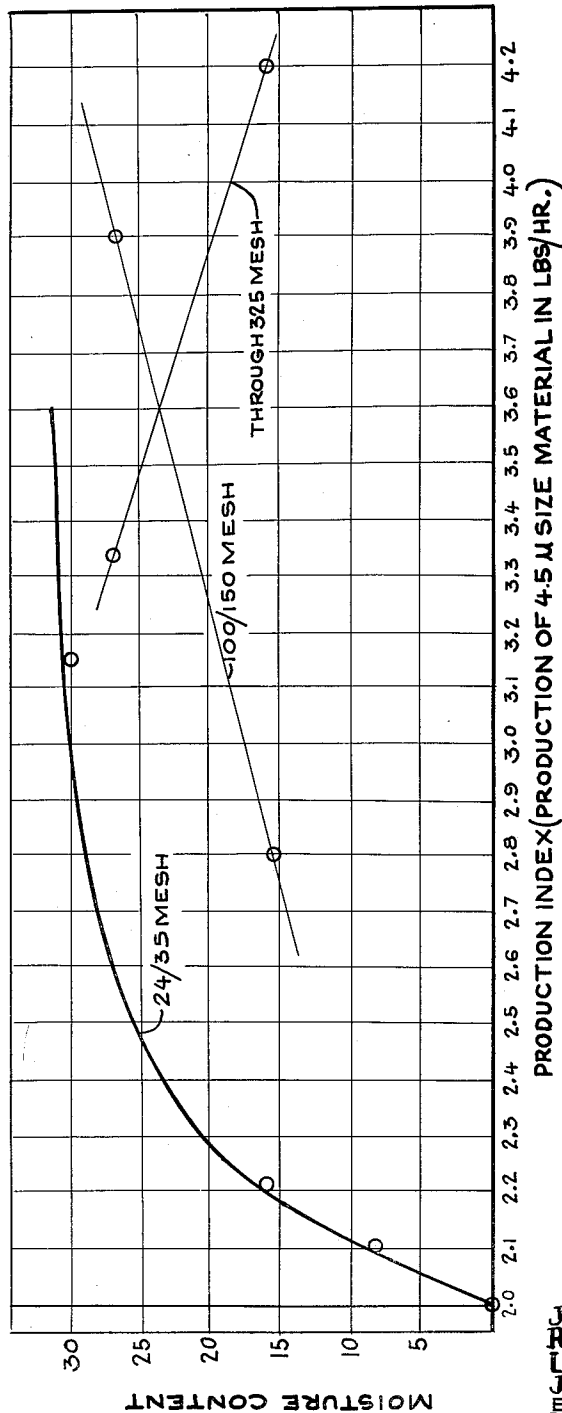


Fig. 3

INVENTORS
Jean M. Bourguet
Robert D. Drew
Louis P. Evans
Jonas P. Harrison

BY
Raymond W. Barclay
ATTORNEY

1

2,983,453

METHOD OF PULVERIZATION

Jean M. Bourguet, Woodbury, Robert D. Drew, Wenonah, and Louis P. Evans and Jonas P. Harrison, Woodbury, N.J., assignors to Socony Mobil Oil Company, Inc., a corporation of New York

Filed Mar. 13, 1958, Ser. No. 721,162

6 Claims. (Cl. 241-1)

This invention relates to an improved method for effecting pulverization of fragmented solid porous friable material. More particularly, the present invention is concerned with a method for increasing the capacity or throughput of a pulverizer wherein solid pulverulent feed material in the form of small particles is ground to a predetermined size by the action of jets of gaseous fluid introduced at superatmospheric pressure into a confined zone containing said material, causing the particles of said material to be entrained in the atmosphere of said gaseous fluid and to be recirculated to such jets until reduced to the desired degree of fineness.

Pulverizers of the type, to which the present invention is applicable, involve those adapted for pulverizing solid materials by fluid jet action. In such pulverizers, jets of gaseous fluid are introduced at high velocity into an annular grinding zone, which grinding zone contains the material to be pulverized in the form of discrete small particles. An atmosphere of gaseous fluid is produced and maintained as the consequence of the operation of the jets, generally with superheated steam or compressed air. When the nature of the material to be ground requires it, other compressed gases such as for example carbon dioxide or nitrogen may be used. These gaseous fluids, inclusive of such vapors as may be present, inspire the particles of solid material which are to be ground and cause them to be suspended in the streams of gaseous fluid and particles so created.

The high velocity fluid jets are generally directed forwardly in one direction of rotation and effect a reduction of particle size of the material under treatment by impact and attrition of particles against one another and against the chamber walls enclosing the grinding zone, while at the same time causing the charge of material to circulate rapidly within the grinding zone in the direction of the circulating fluid. Due to the rotational effects resulting from the rapid circulation of the charge within the grinding zone, the larger particles are caused to concentrate in such zone where they come repeatedly under the influence of the introduced high velocity jets and are thus repeatedly subjected to the size reducing action produced. The material, sufficiently reduced below predetermined size ranges, is continuously exhausted from the grinding zone by entrainment in the gaseous fluid leaving the same. Comminution and grinding, as indicated, take place to a considerable degree in such pulverizing operation, but the efficiency thereof may be improved in accordance with the present invention to provide a much more effective rate of grinding with the attainment of particles of extremely minute sizes.

It is accordingly an object of the present invention

2

to improve the efficiency of a fluid jet pulverizing operation of the type hereinabove described. Another object of the invention is to increase the capacity of a pulverizing apparatus utilizing fluid jet action to afford a high throughput of material undergoing treatment. A further object is to provide a method for treating a feed charge of solid, porous, pulverulent material by exposing the same to the energy of high velocity fluid jets in a manner to accomplish size reduction of said charge material by the combination of explosive forces, as well as by the aforementioned particle impact and attrition. A very important object of the present invention is the provision of a method for increasing the grinding capacity of a pulverizer of the type described in which the material to be ground is a porous, friable adsorbent solid having a pore volume fraction of at least 0.1. The term "pore volume fraction," as utilized herein, designates the fraction of total volume of a porous solid which is attributable to the volume of the pores therein.

The above and other objects which will be apparent to those skilled in the art are realized in accordance with the method of this invention. Broadly stated, the present invention affords a method for increasing the efficiency of a jet pulverizer to achieve greater throughput by control of the particle size and moisture content of the solid, porous, pulverulent charge material undergoing treatment in said pulverizer. Thus, it has been discovered, in accordance with the present invention, that the rate in production of finely divided material of predetermined size can unexpectedly be increased by controlling the particle size of the porous charge material to greater than about 200 mesh (Tyler) and by controlling the water content of said porous charge material in an amount not in excess of that corresponding to the pore volume fraction of said material, but sufficient to bring about sudden explosion of the controlled size particles of the solid porous charge material when the same are subjected to an elevated temperature in the grinding zone substantially in excess of the boiling point of water and, generally at least about 50° F. above said boiling point. The contact of the water-containing charge particles with the grinding fluid at such elevated temperature causes the particles to explode due to sudden evaporation of the water from the particles. The explosive forces thus generated, in combination with the normal grinding action of the jet pulverizer has, in accordance with the present invention, been found to result in a considerable increase in throughput of the pulverizer.

In one embodiment, the present invention is directed to a reduction in particle size of partially comminuted porous pulverulent solid having a particle size greater than about 200 mesh (Tyler) in a jet pulverizer by controlling the water content of said solid prior to its introduction to the grinding zone of said pulverizer in an amount of at least about 5 percent by weight but not exceeding an amount equivalent to the pore volume fraction of the porous solid and thereafter introducing the solid of such controlled moisture content to the grinding zone of said pulverizer maintained at a temperature sufficiently high to effect sudden evaporation of the water from the solid particles with accompanying explosive shattering of said particles.

In another embodiment, the present invention is di-

rected to a method for effectively grinding to an average particle size of less than 10 microns, a solid, porous, friable material having an initial particle size in the approximate range of 2 to 200 mesh (Tyler) and a pore volume fraction in excess of about 0.1 in a pulverizer utilizing fluid jet action and provided with a grinding zone maintained at least 50° F. above the boiling point of water by controlling the water content of such charge material to an amount of at least 10 percent by weight but insufficient to produce an aqueous slurry of said charge material.

In a further embodiment, this invention is directed to a method for increasing the throughput of a porous, friable, particle-form solid in a jet pulverizer wherein such solid is subjected to the grinding action of high velocity jets of gaseous fluid at a temperature generally in the range of 300° F. to 600° F. by conducting to said pulverizer a charge of said solid of 2 to 200 mesh size (Tyler), suspended in a stream of said fluid and having a minimum water content of 5 percent by weight.

In a still further embodiment, this invention is directed to an improved method for grinding a porous inorganic oxide gel characterized by an initial particle size of between about 2 and about 200 mesh (Tyler) and a pore volume fraction in the approximate range of 0.1 to 0.9 and a water content between about 10 and about 90 percent by weight by subjecting the same to the grinding action of high velocity jets of steam in a confined zone of a jet pulverizer maintained at a temperature between about 300° F. and about 600° F. to effect reduction in the average size of said introduced gel particles to less than about 10 microns.

The charge material undergoing pulverization in accordance with this invention is a porous, friable solid, generally characterized by a pore volume fraction in excess of about 0.1. It is contemplated that any solid materials meeting the above definition may be effectively treated by the method of the invention. Representative materials include clays, activated carbon, porous metals, porous glasses, gelatinous precipitates, gels and the like. Inorganic oxide gels are particularly suitable as feed materials for pulverization in accordance with the method described herein. The particular chemical composition of the gel feed or other material does not appear to be critical. Thus, gels of silica, alumina, molybdena, magnesia, chromia, zirconia, thoria, titania, manganese oxide and composites thereof are suitably pulverized by the present method. The resulting pulverized material generally having an average particle size of less than 10 microns and more usually in the range of 1 to 5 microns is useful in the form of or in the manufacture of catalysts, catalyst supports, adsorbents, desiccants, and zeolites.

On the other hand, the physical characteristics of the charge material are important to the success of the method of the invention. Thus, the charge material should necessarily be of a porous pulverulent nature and have sufficient porosity such that the quantity of adsorbed water will upon sudden evaporation thereof upon exposure to elevated temperatures prevailing in the grinding chamber be sufficient to cause explosive shattering of such material. The material undergoing pulverization generally has a pore volume fraction greater than about 0.1 and more usually within the approximate range of 0.1 to 0.9. The particle size of the porous charge material should necessarily be larger than about 200 mesh (Tyler) and generally within the approximate range of 2 to 200 mesh (Tyler). A screen of approximately three-eighths inch opening corresponds to a 2 mesh screen (Tyler). It has been established, as will be evident from data presented hereinafter, that if the particle size of the porous charge material is finer than about 200 mesh, the effect of water content of the charge material on the rate of throughput of such material in the jet pulverizer unexpectedly has a reversed or opposite effect from that ob-

tainable by following the method of the invention. Thus, it has been established, in accordance with the present invention, that an increased throughput of porous pulverulent solid material may be achieved in a jet pulverizer with an increasing water content of such material provided that the particle size of the feed material to such pulverizer is maintained greater than about 200 mesh (Tyler). When the particle size of the feed material is finer than about 200 mesh (Tyler), it has been observed that the throughput of such material unexpectedly decreased with an increasing water content. It is accordingly an essential and critical feature of the invention that the particle size of feed material be greater than about 200 mesh (Tyler). Under normal conditions of operation, the particle size of the feed will be in the approximate range of 2 to 200 mesh (Tyler). The reason for the above noted reversal phenomena is not known with certainty. It would appear, however, without being limited by any theory that particles of feed material finer than about 200 mesh in the presence of adsorbed water have the tendency to cohere or stick together requiring a further grinding effort to obtain the desired product of predetermined fine size and thereby imposing a greater burden on the jet pulverizer with the consequent observed decrease in the rate of throughput.

The water content of the particle-form charge material is necessarily sufficient to effect explosive shattering of the particles due to sudden evaporation of such water upon contact with the gaseous fluid at the elevated temperature prevailing in the grinding chamber of the jet pulverizer. Generally, the amount of water present is at least about 5 percent by weight but not in excess of an amount of water corresponding to the pore volume fraction of the charge material. More particularly, the minimum water content of the charge material will be about 10 percent by weight, usually within the approximate range of 10 to 90 percent by weight and preferably between about 20 and about 40 percent by weight. It will be understood that the amount of water in each instance, however, is less than an amount equivalent to the pore volume fraction of the charge material so that all of the water present is in an adsorbed state. An aqueous slurry of the particle-form charge material, for example, has not been found to be suitable for accomplishment of the objectives of the invention since the particles in the form of such slurry are not amenable to the sudden evaporation of water with accompanying explosive shattering such as achieved in the method of the invention. The water content of the charge material can be controlled in any suitable manner either by drying the charge material to the requisite water content or by the addition of water to the charge material before the same enters the grinding chamber of the jet pulverizer, for example, by the addition of water to the feed line entering such chamber. The water may be added in a controlled amount in the form of a spray or as a continuous stream of liquid mixed in the feed line with the particle-form charge. Alternatively, the particles of charge material may be contacted, either on a static or continuous basis, with highly humidified air to afford a product of the requisite water content.

The particle-form charge material containing adsorbed water then enters the grinding chamber of a jet pulverizer, suitably suspended in a stream of the gaseous grinding fluid at a high velocity where it is thrown in the radial path of the grinding fluid entering said chamber at a temperature above the boiling point of water and generally at a temperature sufficiently high to effect sudden evaporation of the adsorbed water with accompanying explosive shattering of the particle charge but at a temperature below that at which sintering or fusion of the charge material is encountered. Usually, the temperature of the fluid entering the grinding chamber is within the range of about 300° F. to about 600° F. but may vary outside the range depending on the character of the specific

grinding fluid being employed as well as on the type and nature of the charge material undergoing treatment. As indicated hereinabove, the grinding fluid employed will generally but not necessarily be either steam or compressed air.

Upon contact of the water-containing charge material and high temperature gaseous fluid in the grinding chamber, the particles of said material undergo an explosive shattering to fine pieces which are thereafter subjected to further impact and attrition in the grinding chamber as a result of the rotational action of the high velocity jets of gaseous fluid and such action is thereafter maintained until the charge material is reduced to a size of predetermined fineness. As a consequence of the initial aforesaid explosive shattering effect, the rate of production of desired finely ground product can be unexpectedly and greatly increased resulting in a much more efficient pulverizing operation over that in which such initial explosive shattering effect is absent.

The method of the invention may be more readily understood by reference to the attached drawing wherein:

Fig. 1 depicts, in schematic form, suitable procedure and jet pulverizer to which the method of the invention relates.

Fig. 2 shows, in schematic form, a plan view of the grinding chamber of the jet pulverizer shown in Fig. 1.

Fig. 3 shows the effect of moisture content of the charge material on the rate of production of ground product for varying particle size charge material.

Turning now to Figs. 1 and 2 of the drawing, fresh feed in the form of particles having a size greater than about 200 mesh (Tyler) and generally in the range of 2 to 200 mesh (Tyler) is introduced into humidifying chamber 10 wherein a counter-flowing stream of humidified air is introduced through conduit 11 and withdrawn through conduit 12. The respective rates of flow of charge material and humidified air are such that the water content of the feed material leaving the humidifying chamber is within a predetermined range, generally at least about 5 percent and more usually between 10 and about 90 percent by weight. The particle-form material containing adsorbed water passes from the humidifying chamber through conduit 13 and is suitably suspended therein in a stream of gaseous fluid, desirably the same as that utilized for grinding, injected through inlet 14. The suspended particles are then introduced to grinding chamber 15 where the water-containing particles come into contact with the grinding fluid, for example, steam at a temperature of approximately 450° F. and at a pressure of 5 to 10 p.s.i.g. and introduced to said grinding chamber through a plurality of jets 16, only two of which have been shown for purposes of simplicity, evenly spaced along the periphery of the grinding chamber. The grinding fluid, such as air or steam, may be introduced to the grinding chamber at a temperature of about 600° F. and under a pressure of about 150 pounds per square inch gauge. Upon coming into contact with the high temperature grinding fluid in the grinding chamber, the water containing charge particles undergo initial explosive shattering and are thereafter further ground to predetermined particle size by the action of the high velocity jets of grinding fluid. The finely ground product passes from the grinding chamber to surge chamber 17 in communication therewith and thereafter passes upwardly, suspended in an atmosphere of the grinding fluid, through outlet 18. The process of the invention is, of course, not to be limited to the above specific apparatus or operational details. Thus, in some instances or apparatus, it may be desirable, for example, to withdraw the finished ground product through an outlet projecting downwardly from chamber 17.

The following comparative examples will serve to illustrate the method of the invention and particularly the effect of moisture content of the charge material on the rate of production of ground product.

Example 1

Silica-alumina gel containing a small amount of chromia was prepared by mixing streams comprising: (1) aluminum sulphate dissolved in an aqueous solution of sulphuric acid, and (2) sodium silicate containing powdered dried silica-alumina gel of specified particle size. The sulphuric acid-aluminum sulphate stream was composed of 3.79 pounds of 96.3 percent sulphuric acid, 7.74 pounds of aluminum sulphate (containing 43 percent weight water), 0.315 pound of potassium chrome alum, and 117 pounds of water. The sodium silicate stream containing silica-alumina gel powder was composed of 48.0 pounds of sodium silicate having an Na_2O to SiO_2 ratio of 1:3.22 and a density of 41° Bé., 48.4 pounds of water, and 3.6 pounds of dried silica-alumina gel powder containing approximately 90 percent weight SiO_2 and 10 percent weight Al_2O_3 , and having a particle diameter of approximately 2.0 microns.

Four hundred fifty-six cc./min. of the sulphuric acid-aluminum sulphate and 448 cc./min. of the sodium silicate slurry were mixed in a mixing nozzle to form a sol, which set to a hydrogel in 2.6 seconds at 60° F. The sol was formed into spheroidal hydrogel beads.

The resulting hydrogel particles, having a pH of 8.5 were treated for 12 hours at 69° F. while covered with water. The hydrogel particles were thereafter base-exchanged with an aluminum sulphate solution to remove sodium from the structure of the gel, washed free of soluble salts, and dried in superheated steam at 260° F. for 6 hours.

Example 2

Gel product prepared as in Example 1 and containing 8 percent by weight moisture, as received from the dryer, was preground to pass through a 24 mesh but be retained on a 35 mesh Tyler sieve, i.e. 24/35 mesh size and thereafter pulverized in a 2" jet pulverizer of the type shown in Figs. 1 and 2, utilizing high velocity jets of air at a temperature of about 600° F. The production of 4.5 micron size material was about 2.1 pounds per hour.

Example 3

Gel product prepared as in Example 1 was preground to 24/35 mesh size and thereafter tempered in air at 1300° F. until free of moisture. The resulting material was then introduced into a jet pulverizer under conditions identical with those employed in Example 2. The production of 4.5 micron size material decreased 5 percent from that of Example 2.

Example 4

Gel product prepared as in Example 1 was preground to 24/35 mesh size and thereafter humidified by treatment in humidified air to a water content of 16 percent by weight. The resulting material was then introduced into a jet pulverizer under conditions identical with those of Example 2. The production of 4.5 micron size material in this instance increased 5 percent over that of Example 2.

Example 5

Gel product prepared as in Example 1 was preground to 24/35 mesh size and thereafter humidified by treatment in humidified air to a water content of 30 percent by weight. The resulting material was then introduced into a jet pulverizer under conditions identical with those of Example 2. The production of 4.5 micron size material in this instance increased 50 percent over that of Example 2.

Example 6

Gel product prepared, as in Example 1, was preground to pass through a 100 mesh but be retained on a 150 mesh Tyler sieve, i.e. 100/150 mesh and thereafter humidified by treatment in humidified air to a water content of 27 percent by weight. The resulting material was

7

then introduced into a jet pulverizer under conditions identical with those of Example 2. The production of 4.5 micron size material was about 3.9 pounds per hour.

Example 7

Gel product prepared as in Example 1 was preground to 100/150 mesh and humidified in air to a water content of 16 percent by weight. The resulting material after pulverizing under conditions identical with those of Example 2, showed a production of 4.5 micron size material of about 2.8 pounds per hour.

Example 8

Gel product prepared as in Example 1 was preground to a particle size smaller than 325 mesh and thereafter humidified to a water content of 27 percent by weight. The resulting material after pulverizing under conditions identical with those of Example 2 showed a production of about 3.35 pounds per hour.

Example 9

Gel product identical with that of Example 8 was humidified to a water content of 16 percent by weight. The resulting material after pulverizing under conditions identical with those of Example 8 showed the production of 4.5 micron size material of about 4.2 pounds per hour.

The results of the foregoing illustrative examples are shown graphically in Figure 3. It will be noted from such figure that the production index i.e. the production of 4.5 micron size material in pounds per hour increases with increasing moisture content utilizing a charge material having a particle size of 24/35 mesh. Likewise, the production index for charge material having a particle size of 100/150 mesh increased with an increase in moisture content. The charge material having a particle size smaller than 325 mesh, however, gave a smaller production index with increasing moisture content indicating that the rate of throughput of such charge material in the jet pulverizer decreased as the water content thereof increased. This, as will be apparent, is the exact opposite of the effect observed with charge material having a particle size of 24/35 mesh and 100/150 mesh. It is accordingly essential to the success of the method of the invention that the particle size of the charge material be larger than about 200 mesh.

It is to be understood that the above description is merely illustrative of preferred embodiments of the invention of which many variations may be made by those skilled in the art without departing from the spirit thereof.

We claim:

1. A method for effecting a reduction in particle size of partially comminuted porous pulverulent solid having a particle size greater than about 200 mesh in a jet pulverizer wherein said solid is subjected to the grinding action of high velocity jets of gaseous fluid in a confined zone which comprises controlling the water content of said solid prior to its introduction to the grinding zone of said pulverizer in an amount of at least about 5 percent by weight but not exceeding an amount equivalent to the pore volume fraction of the porous solid, introducing the solid of such controlled moisture content to the grinding zone of said pulverizer maintained at a temperature sufficiently high to effect sudden evaporation of the water from the solid particles with accompanying explosive shattering of said particles and thereafter subjecting the shattered particles to impact and attrition resulting from turbulence attributable to the rotational action of said high velocity jets until the particle size of said solid is reduced to a predetermined degree of fineness.

2. A method for increasing the throughput of a porous, friable, particle-form solid feed material having a pore volume fraction of at least 0.1 in a jet pulverizer wherein said feed material is subjected to the grinding action of high velocity jets of gaseous fluid in a confined zone maintained at an elevated temperature in substantial excess of

8

the boiling point of water, causing the particles of said material to be entrained in the atmosphere of said gaseous fluid and to be recirculated to such jets until reduced to a predetermined degree of fineness which comprises introducing to said pulverizer, particles of said material of a size greater than about 200 mesh characterized by a water content in an amount not exceeding that corresponding to the pore volume fraction of said material but sufficient to effect explosive shattering of said particles upon contact with said gaseous fluid maintained at said elevated temperature in said grinding zone.

3. A method for increasing the throughput in a jet pulverizer of a porous, friable, particle-form solid feed material having a pore volume of at least 0.1 wherein said feed material is subjected to the grinding action of high velocity jets of gaseous fluid in a confined zone maintained at a temperature at least 50° F. above the boiling point of water, causing the particles of said material to be entrained in the atmosphere of said gaseous fluid and to be recirculated to said jets until reduced to a predetermined degree of fineness which comprises introducing to said pulverizer, particles of said material of a size between about 2 and about 200 mesh characterized by a water content of at least about 5 percent by weight but not in excess of an amount corresponding to the pore volume fraction of said material.

4. A method for effecting a reduction in the particle size of inorganic oxide gel particles characterized by an initial particle size of between about 2 and about 200 mesh and a pore volume fraction in the approximate range of 0.1 to 0.9 and a water content between about 10 and about 90 percent by weight which comprises introducing said particles of gel to a jet pulverizer wherein said gel particles are subjected to the grinding action of high velocity jets of gaseous fluid in a confined zone maintained at a temperature between about 300 and about 600° F. to effect reduction in the average size of said introduced gel particles to less than 10 microns.

5. A method for effecting a reduction in particle size of partially comminuted porous pulverulent adsorbent solid having a particle size in the approximate range of 2 to 200 mesh in a jet pulverizer wherein said solid is subjected to the grinding action of high velocity jets of gaseous fluid in a confined zone which comprises controlling the water content of said solid prior to its introduction to the grinding zone of said pulverizer in an amount of at least about 5 percent by weight but not exceeding an amount equivalent to the pore volume fraction of the porous solid, introducing the solid of such controlled moisture content to the grinding zone of said pulverizer maintained at a temperature within the approximate range of 300 to 600° F. thereby effecting explosive shattering of said particles due to the sudden evaporation of the water from said particles and thereafter subjecting the shattered particles to impact and attrition attributable to the rotational action of said high velocity jets until said solid is reduced to a predetermined degree of fineness.

6. A method for increasing the throughput of a porous inorganic oxide gel having a pore volume fraction in the range of 0.1 to 0.9 in a jet pulverizer wherein said gel is subjected to the grinding action of high velocity jets of gaseous fluid in a confined zone maintained at an elevated temperature in the approximate range of 300 to 600° F. which comprises introducing to said pulverizer, particles of said gel of a size within the approximate range of 2 to 200 mesh characterized by a water content between about 20 and about 40 percent by weight but not exceeding an amount corresponding to the pore volume fraction of said gel thereby effecting explosive shattering of said gel particles upon contact with said gaseous fluid maintained at said elevated temperature in said confined zone and thereafter subjecting the shattered gel particles to impact and attrition attributable to the rotational action of said high velocity jets to yield a resulting product

having an average particle size within the range of 1 to 5 microns.

References Cited in the file of this patent

UNITED STATES PATENTS

2,032,827	Andrews -----	Mar. 3, 1936
2,392,866	Stephanoff -----	Jan. 15, 1946
2,393,783	Kridel -----	Jan. 29, 1946
2,515,541	Yellott -----	July 18, 1950
2,636,688	Singh -----	Apr. 28, 1953
2,856,268	Young -----	Oct. 14, 1958

FOREIGN PATENTS

528,163 Great Britain ----- Oct. 23, 1940

OTHER REFERENCES

5 Ultra-Fine Grinding and Classification with Fluid Jet
Pulverizers, by M. F. Dufour and J. B. Chatelain, Mining
Engineering, March 1952, pages 262-264.
10 Comparison of Fine . . . Sieves," R.I. 3766, United
States Bureau of Mines, July 1944, page 3.