

March 31, 1953

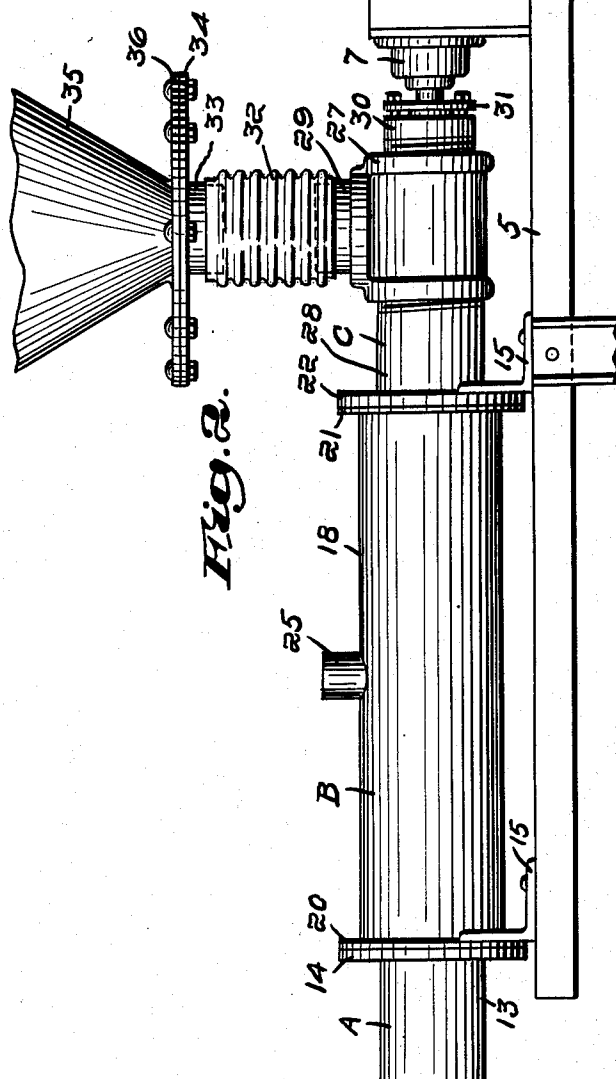
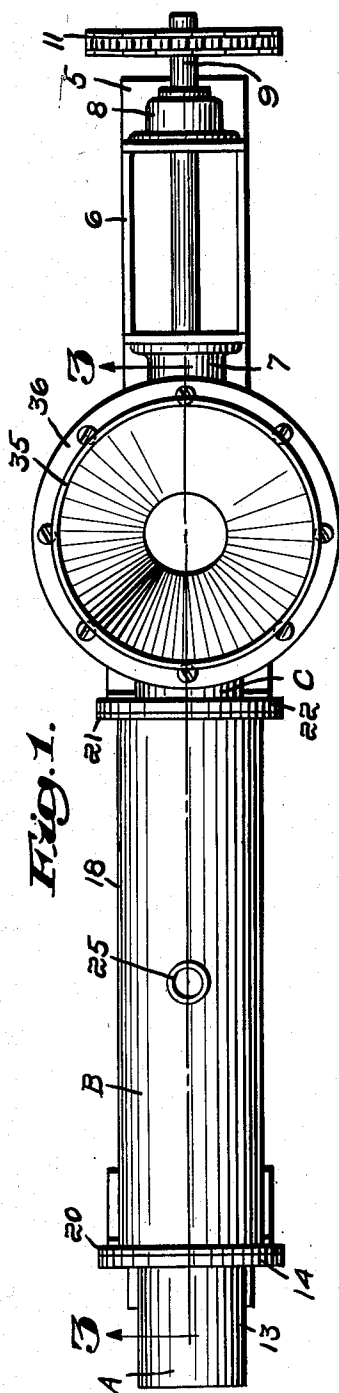
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2,633,074

APPARATUS FOR DENSIFYING DRY POWDERED SOLIDS

Filed May 1, 1948

2 SHEETS—SHEET 1



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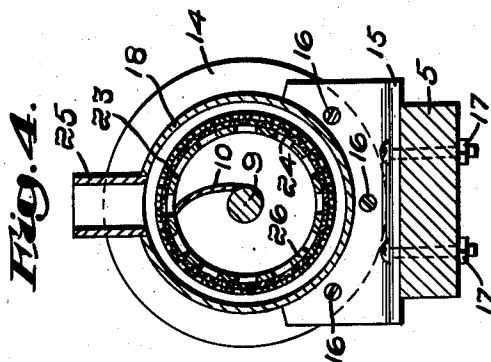
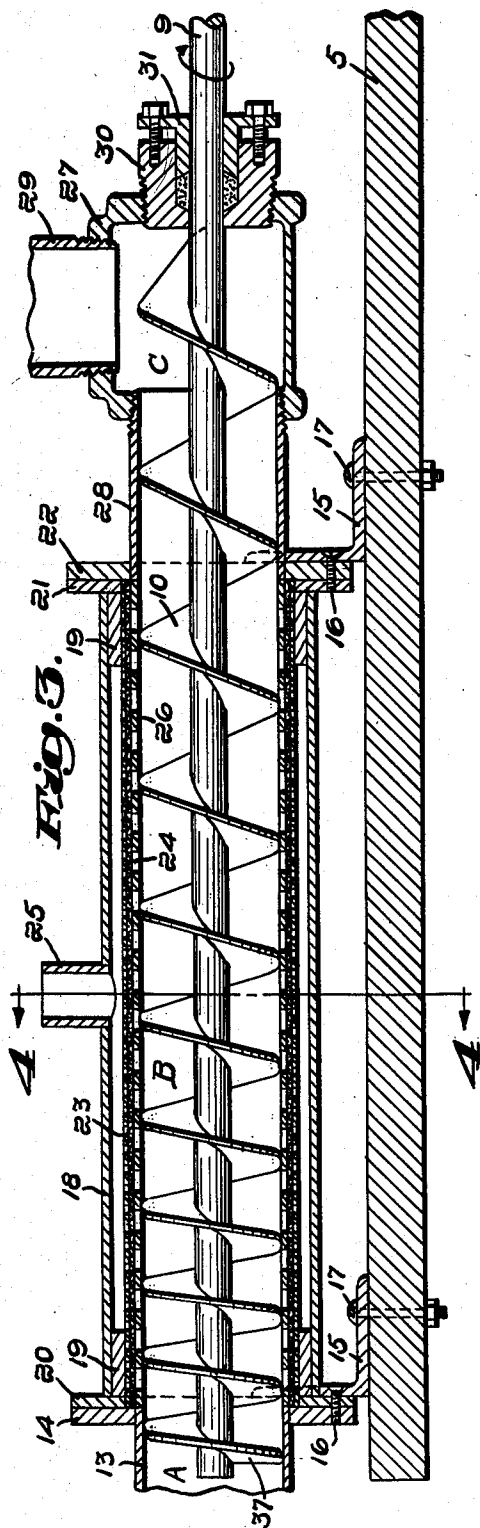
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APPARATUS FOR DENSIFYING DRY POWDERED SOLIDS

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2 SHEETS—SHEET 2



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UNITED STATES PATENT OFFICE

2,633,074

APPARATUS FOR DENSIFYING DRY
POWDERED SOLIDS

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The present invention relates to apparatus for materially increasing the density of powdered substantially dry solids which contain relatively large quantities of occluded or included air and/or other gases. The invention more particularly relates to apparatus for materially increasing the density of finely divided or powdered silica aerogels and other similar aerogels which contain relatively large quantities of occluded or included air and/or other gases as a result of an antecedent pulverizing or grinding operation.

Silica aerogels, for example, those having extremely fine particles, often of microscopic size, are employed as flattening agents, pigments and the like in lacquer, paint and other coating compositions. In order to reduce the aerogel particles to the proper size consistent with such uses, it is necessary to subject the silica aerogel to a comminuting, grinding or pulverizing operation. However, when silica aerogel is pulverized or ground to the necessary degree of fineness, relatively large quantities of air and/or other gases become occluded or included therein and the resulting product has a very low density and is almost impossible to densify by vibrating means. Moreover, it is extremely difficult to densify or compress this material economically and efficiently. On the other hand it is desirable to compress the material without causing the individual particles to agglomerate. Agglomeration of the particles is considered to be undesirable since the material loses some of its hiding power and flattening efficiency. If, during densification, the particles are caused to agglomerate they can be redispersed, but usually only by a prolonged and uneconomical grinding operation.

In the manufacture of carbon black as, for example, lampblack, it is not generally necessary to pulverize or comminute the product since it is generally of the proper particle size as produced. However, relatively large quantities of air and/or other gases are generally occluded or included in the material as a result of the manufacturing operation. Such products and other powdered solids of substantially similar characteristics generally have a low density or low weight per unit of volume, which in some instances may be as low as 1.0 pound per cubic foot, the particular density in any case depending upon the manner in which the material has been manufactured, pulverized or ground and the nature of the material itself.

Because of the low density of such materials it is necessary to compress or densify them to as high a degree as is possible and practical prior to packaging and shipping otherwise the cost of

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packaging and shipping such materials would be prohibitive. However, the densification of such materials should be preferably accomplished without causing substantial agglomeration of the individual particles and in such a manner that the densified material is readily disintegrated into individual particles having substantially the same size as the original particles prior to densification. The present invention affords a simple, efficient and practical method and apparatus for producing such densified materials.

It is, accordingly, one object of the invention to provide a simple and efficient apparatus for increasing the density of substantially dry powdered solid materials which contain relatively large quantities of occluded and included air and/or other gases whereby a product is obtained which is economical to ship and package and which is easily disintegrated into its individual particles.

A further object of the invention is to compress and densify by a continuous method finely divided or pulverized silica aerogels and other pulverized aerogels containing relatively large quantities of occluded air and/or other gases without substantially agglomerating the individual particles thereof.

A further object of the invention is to provide an apparatus for increasing the density of powdered silica aerogels and other powdered aerogels and preparing a product having the characteristics described in the foregoing objects.

Other objects and advantages of the invention will appear from the following description when taken in connection with the accompanying drawings and the appended claims.

The invention is carried out, in general, by subjecting a powdered or finely divided substantially dry solid material as, for example, a pulverized silica aerogel which contains relatively large quantities of occluded or included air and/or other gases to the simultaneous or concurrent action of a compressive force, preferably progressively increasing compressive force, and sub-atmospheric pressure as exemplified by suction, vacuum or the like, while confining such powdered material in a substantially fixed volume enclosure, and then removing the compressed or densified material from the zone of compressive force and sub-atmospheric pressure after the material has been densified or compressed to the desired extent. In treating the materials as described above, a compression device of the type hereinafter described is preferably employed.

A variety of powdered and substantially dry solid materials can be densified according to the practice of the invention as described herein. Thus, the lampblack which is prepared by the controlled oxidation or combustion of oil can be densified immediately after it is collected by the method and apparatus herein described. Other substantially dry powdered solid materials such as dry and pulverized H-acid, DDT and the like, which materials contain occluded or included air and/or other gases as a result of their method of preparation or as a result of a subsequent pulverizing, grinding or comminuting operation, may also be successfully densified by the practice of the present invention. The present invention is particularly directed to the densification of pulverized or powdered, substantially dry silica aerogels and commercial lampblacks which contain relatively large quantities of occluded or included air and/or other gases, and, for convenience, the invention will be described with reference to the densification of such material.

Pulverized silica aerogels of the type hereinbefore described may be prepared in a variety of ways. In general, a silica aerogel having relatively coarse particles is prepared initially and is then pulverized or comminuted in a suitable apparatus such as a hammer mill, ball mill, air attrition mill or the like until particles of the desired particle size are obtained. Such silica aerogel may be prepared, for example, by first reacting an aqueous alkaline silicate solution such as aqueous sodium silicate and an inorganic acid such as sulfuric acid to form a silica gel. This gel is first washed substantially free of inorganic salts or electrolytes and is then heated above the critical pressure of the liquid in the gel, which liquid may be water or some water-miscible organic liquid such as ethanol which has been employed to replace the water in the gel. The foregoing operation is carried out in a fixed volume system such as an autoclave and the heating is continued until substantially all of the liquid is converted to the vapor phase. The vaporized liquid is then slowly released from the fixed volume system at such a rate that the gel structure is not materially altered, that is, the gel occupies substantially the same volume as the gel originally placed in the system. The foregoing method is described in detail in the Kistler Patents Nos. 2,093,454; 2,188,007 and 2,249,767. The product so obtained is usually in lump form after removal from the autoclave and is then ground or pulverized to the requisite particle size as hereinbefore described.

A similar silica aerogel can be prepared by autoclaving a suitable acid-reacting silica organo-aquasol instead of a silica gel of the type described above. The procedure for autoclaving such an organo-aquasol is essentially the same as for the autoclaving of the silica gel. Such an acid-reacting silica organo-aquasol can be prepared by first reacting an alkaline silicate such as sodium silicate and an inorganic acid such as sulfuric acid and then adding thereto a water-miscible organic solvent such as ethanol or isopropanol in sufficient quantity to precipitate a substantial proportion of the electrolyte formed during such reaction. The precipitated electrolyte is then removed as by filtration, centrifuging or the like. Such organo-aquasol and the method of preparing same is described in greater detail in the Marshall Patent No. 2,285,449.

The silica aerogels prepared as described above are adapted to be ground into very fine powders having a majority of particles with a particle size

of 10 microns or less. In this state they are quite fluffy and contain relatively large quantities of occluded or included air. If they are compressed they tend to return to their original volume when the pressure is released. Such powdered silica aerogels have a density varying, in general, between about 1.0 pound to 4 pounds per cubic foot and may be densified by the method and apparatus described herein to any practical density as, for example, a density of 5 to 9 pounds per cubic foot.

In the manufacture of lampblack by the controlled oxidation or combustion of oil, the product as obtained generally has a density of from about 4 to 8 pounds per cubic foot depending upon the method of preparation and the oil used. By the practice of the invention the density of such product can be increased to about 8 to 30 pounds per cubic foot without substantially causing the individual particles to agglomerate.

It is also possible by the practice of the present invention to continuously densify fluffy materials such as the above and to obtain a product which is substantially uniform in density irrespective of normal fluctuations in density of the material to be densified.

The accompanying drawings illustrate a preferred embodiment of the apparatus used in preparing such densified products, in which—

Figure 1 represents a top plan view showing one embodiment of a screw compressor constructed in accordance with the present invention,

Figure 2 is a side elevation thereof,

Figure 3 is an enlarged longitudinal sectional view of the screw compressor casing or assembly taken along line 3—3 of Figure 1, and

Figure 4 is an enlarged cross sectional view taken along line 4—4 of Figure 3.

Referring to the drawings, wherein similar numerals refer to similar parts, part 5 is a base for the screw compressor, which base may be fixed or mounted on wheels or casters or the like and is thus adapted to be moved from one position to another. Firmly secured to base 5 is a housing 6 on which is mounted a roller bearing assembly 7 and a ball bearing assembly 8. Screw shaft 9 carrying the screw 10 is mounted to rotate within the bearings 7 and 8 and has a chain driven sprocket 11 keyed thereto. Sprocket 11 is driven by chain 12 through motor driven sprocket (not shown) or any other suitable device. The screw 10 which is securely fastened to shaft 9 may be a variable pitch screw as shown in Figure 3 or may be a screw of equal pitch, although the construction shown is preferred.

Referring specifically to Figures 3 and 4, which show the screw compressor assembly and casing therefor, section A is the discharge section comprising a cylindrical steel shell 13 and an annular steel flange 14 welded thereto, section B is the vacuum section, shown between the dotted lines in Figure 3 and section C is the feed section, where the dry powdered material to be compressed is fed to the apparatus. The entire assembly is firmly secured to angle irons 15 by screws 16, and the angle irons 15 are in turn securely bolted to base 5 by stove bolts 17.

The vacuum section B, as shown in Figures 3 and 4, comprises an outer cylindrical steel shell 18 having internally mounted annular collars 19 and externally mounted annular flanges 20 and 21 all welded thereto to form a firm unitary structure. Annular flange 20 is

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bolted to flange 14 of discharge section A and flange 21 is bolted to flange 22 of feed section C making the entire casing firm and unitary. Mounted within cylindrical shell 18 and welded to annular collars 19 is a perforated cylindrical steel plate 23 having a large number of relatively small perforations. If desired this may be replaced by a strong cylindrical wire screen wrapped with relatively close helical windings of heavy steel wire having a sufficient diameter to prevent the wire screen from contacting the shell 18 when dry powdered material is conveyed through the vacuum section B by screw 10. Within the perforated cylindrical steel plate 23 is a cylindrically shaped canvas cloth 24 or other suitable filtering cloth or medium which is of such construction as to substantially prevent penetration of dry powdered material there-through. Fabric 24 provides a lapped fit with perforated plate 23 when air is exhausted from the vacuum section B through pipe 25 by means of a suitable exhaust or vacuum system (not shown). Freely mounted within the cylindrical canvas cloth 24 and abutting on the inner ends of casing section A and B is a cylindrical perforated thin steel sheet 26 having relatively large perforations therein as compared to perforated cylindrical plate 23. This cylindrical perforated sheet may be replaced by a relatively light steel wire screen or may be omitted altogether, since it is employed primarily to prevent excessive wear on canvas cloth 24 by the rotating action of screw 10 and the abrasive effect of the dry powdered material being compressed. The inner surface of the perforated cylinder 26 forms a substantially continuous surface with the inner surface of the discharge section A and feed section C.

The feed section C as shown in Figures 1, 2 and 3 comprises an internally threaded T 27 in which is screwed externally threaded cylindrical steel shells 28 and 29 and a standard stuffing box assembly 30 having a metal bushing 31 which seals the feed end of the screw compressor assembly and substantially prevents outside air from leaking into the assembly along shaft 9 when air is exhausted from the assembly through pipe 25. Cylindrical steel shell 28 is welded or otherwise firmly connected to annular flange 22. In order to facilitate the loading of the screw compressor, the cylindrical shell 29 is connected through a flexible rubber hose 32 or the like to a similar steel shell 33 having an annular drilled flange 34 welded thereto. Positioned above flange 34 is a hopper 35 equipped with an annular drilled flange 36 similar to flange 34, which flanges may be screwed together as shown in Figures 1 and 2 to give a relatively air-tight fit.

As is shown in Figures 3 and 4, screw 10 rotates within the screw compressor casing and is so constructed and arranged as to provide a close fit with the inner surfaces of the fabric 24 or the cylinder 26. The clearance actually used is preferably of the order of 0.01 to 0.2 inch and in general should be such as to substantially prevent the build-up of layers of the powdered material between the outer edges of the screw 10 and the inner walls of the fabric 24 or cylinder 26. At the same time the clearance should be such as to prevent excessive wear on the screw and the inner walls of the cylinder 26. When cylinder 26 is omitted from the structure, the screw 10 should be in close fitting relationship with canvas cloth 24. The vane 37 at

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the discharge end of the screw 10 rides on the inner wall of steel shell 13 which thus furnishes additional support for the screw shaft 9.

The operation of the screw compressor is as follows:

Dry powdered silica aerogel or other powdered dry material stored in a hopper 35 is fed under gravity into feed section C and this material is forwarded by the rotating screw 10 toward the discharge end of the apparatus. Air is exhausted by means of a suitable vacuum apparatus through pipe 25 and thus a relatively large portion of occluded or included air is removed from the material. Such air passes through the perforated cylindrical steel sheet 26, the canvas fabric 24 and the perforated cylindrical steel plate 23 while the powdered dry material is precluded by virtue of canvas fabric 24 from being exhausted therewith. The compressor reaches its maximum efficiency when the powdered material reaches the discharge section A since a highly effective seal is formed by the powdered material itself and air is substantially prevented from entering into the vacuum section from the discharge end of the compressor. When the air seal is formed at the discharge end in this manner the continued exhaustion of air through pipe 25 creates a suction within the screw compressor casing shown in Figure 3 and this suction continuously drags powdered material from the hopper 35 into the feed section C where it is continuously picked up and forwarded by screw 10. When a variable pitch screw 10, as shown in Figure 3, is used in forwarding the dry powdered material, air is not only exhausted therefrom as described above, but the screw exerts a progressively increasing compressive force on the material and compresses same. The air which is forced out of the material in this manner is exhausted through pipe 25 as described above. When the compressed and densified material reaches the discharge section A and emerges therefrom it is collected in suitable packaging containers (not shown) and is then ready for shipment.

The degree to which the dry powdered material is densified or compressed depends upon several factors such as the speed at which the material is forwarded through the compressor, the order of vacuum or suction applied to the compressor assembly, the nature and density of the material itself, whether or not a variable pitch screw is used and the length of the vacuum section. In general, the faster the screw is rotated the less the powdered material is compressed or densified. Conversely, if the screw is rotated at very low speeds the material may be compressed excessively and may be compacted to such an extent that the discharged product cannot be easily disintegrated into individual particles corresponding to those originally fed into the compressor. The degree to which the system is evacuated or the degree of vacuum employed is not particularly critical. In ordinary operation of the apparatus a vacuum of the order of 5" to 10" of mercury (this corresponds to an absolute pressure of 25" to 20" of mercury) has given satisfactory results in the densification of powdered or pulverized silica aerogels and commercially produced lamp-black.

A further understanding of the invention will be obtained from the following examples which are intended to be illustrative but not limitative of the scope of the invention.

Example I

Dry powdered silica aerogel having a density of about 2 pounds per cubic foot was fed through a device of the construction shown in the accompanying drawings. A variable pitch screw as shown in Figure 3 was employed to convey the material and was rotated at a speed of 60 R. P. M. Air was exhausted through pipe 25 so as to establish an absolute pressure in the vacuum section of 24" of mercury. The material issuing from the apparatus at the rate of 175 pounds per hour had a density of 7 pounds per cubic foot and was readily dispersible in a ball mill with organic solvents, water and the like into substantially the same discrete particles as it contained prior to the densification.

Example II

Dry powdered silica aerogel having a density of about 3 pounds was densified as described in Example I except that the variable pitch screw was rotated at a speed of 100 R. P. M. The material which was packaged as it emerged from the apparatus at the rate of 225 pounds per hour had a density of 8 pounds per cubic foot and was readily dispersible into its original individual discrete particles.

Example III

Dry pulverized silica aerogel having a density of about 4 pounds per cubic foot was fed through an apparatus constructed in accordance with the accompanying drawings. A variable pitch screw as shown in Figure 3 was employed to convey the material and was rotated at a speed of 140 R. P. M. Air was exhausted through pipe 25 by means of a vacuum pump so as to maintain an absolute pressure of 25" of mercury within the vacuum section B of the apparatus. The material issuing from the apparatus at the rate of 300 pounds per hour had a density of about 8.5 pounds per cubic foot and was readily dispersed into its original individual particles.

Example IV

A substantially dry lampblack having a density of about 5 pounds per cubic foot was densified in an apparatus constructed in accordance with the accompanying drawings. The screw conveyor shown in Figure 3 was rotated at 130 R. P. M. and the lampblack was continuously fed into the apparatus. A vacuum pump was employed to exhaust air through pipe 25 and to maintain an absolute pressure of 24" of mercury within vacuum section B of the apparatus. The product which emerged from the discharge section A of the apparatus at the rate of 500 pounds per hour had a density of about 25 pounds per cubic foot and was readily dispersible in a ball mill with liquids such as organic solvents, water and the like into its original individual discrete particles.

Example V

A substantially dry lampblack having a density of about 5 pounds per cubic foot was densified as described in Example IV except that the variable pitch screw was rotated at a speed of 105 R. P. M. The densified product which issued from the apparatus at a rate of 350 pounds per cubic foot had a density of about 19 pounds per cubic foot and could be readily dispersed into its original particle size.

It can be seen from the foregoing description and the above examples that dry powdered ma-

terials can be densified to a varying degree which is dependent upon the density and nature of the material being compressed or densified, the rate of feed and discharge, the extent to which the system is exhausted and upon other factors. The material may be increased in density from about 2 to 6 fold by the practice of the invention.

What is claimed is:

1. A compressing device for densifying substantially dry powdered solids comprising a horizontally positioned casing provided with a rotatable screw conveyor for conveying powdered solids along the longitudinal axis of said casing; a perforated cylindrical insert liner encompassing said screw conveyor in close fitting relationship therewith and loosely mounted in said casing, said liner having large perforations therein; a freely mounted cylindrical shaped filtering medium closely encompassing said liner; a cylindrical screen encompassing said filtering medium and fastened to said casing in spaced relationship with the inner walls of said casing, said screen having a large number of small perforations therein aligned with the large perforations in said liner; said casing having an inlet opening adjacent one end of said casing and being provided at the other end thereof with a cylindrical non-perforated member into which the discharge end of the screw conveyor extends and which forms an extension of said liner; means for rotating said screw conveyor; means for supplying substantially dry powdered solids to the inlet opening in said casing; and means for exhausting air from that part of the casing in which said screen is located.

2. A compressing device for densifying substantially dry powdered solids comprising a horizontally positioned cylindrical casing provided with a rotatable variable pitch screw for conveying powdered solids along the longitudinal axis of the casing, a perforated cylindrical liner encompassing said screw in close fitting relationship therewith and loosely mounted in said casing, said liner having large perforations therein; a freely mounted filtering fabric closely encompassing said liner, which fabric prevents the passage of the solids therethrough; a cylindrical screen encompassing said filtering fabric and fastened to said casing in spaced relationship with the inner walls of the central portion of said casing, said screen having a large number of small perforations therein aligned with the large perforations in said liner; said casing having an inlet opening adjacent one end of said casing and an air outlet in the central portion of said casing, and being provided at the other end thereof with a cylindrical non-perforated member into which said screw extends and which forms an extension of said liner; means for rotating said screw; means for supplying substantially dry powdered solids to the feed opening in said casing; and means for exhausting air from said casing through said air outlet in the casing.

3. A compressing device for densifying and exhausting air from substantially dry powdered solids comprising a horizontally positioned cylindrical casing provided with a rotatable variable pitch screw extending longitudinally through said casing; a perforated cylindrical insert liner encompassing said screw in close fitting relationship therewith and loosely mounted in the central portion of said casing, said liner having large perforations therein; a freely mounted cylindrical-shaped filtering fabric closely encompassing said liner, which fabric prevents the passage

of the solids therethrough; a cylindrical screen encompassing said filtering fabric and fastened to said casing in spaced relationship with the inner walls of the central portion of the casing, said screen having a large number of small perforations therein aligned with the large perforations in said liner; said casing having a feed opening adjacent to the feed end of said screw and an air outlet in the central portion of the casing and being provided with a cylindrical non-perforated member into which the discharge end of the screw extends and which forms an extension of said liner; a container for storing said solids having an outlet near the bottom thereof and positioned above said casing; a hollow member, which is partially flexible, connecting the outlet in said container with the feed opening in said casing providing a substantially air-tight passage extending from said container to the interior of said casing; means for rotating said

screw; and means for exhausting air from said casing through the air outlet in said casing.

WALTER M. DAVIS.

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