

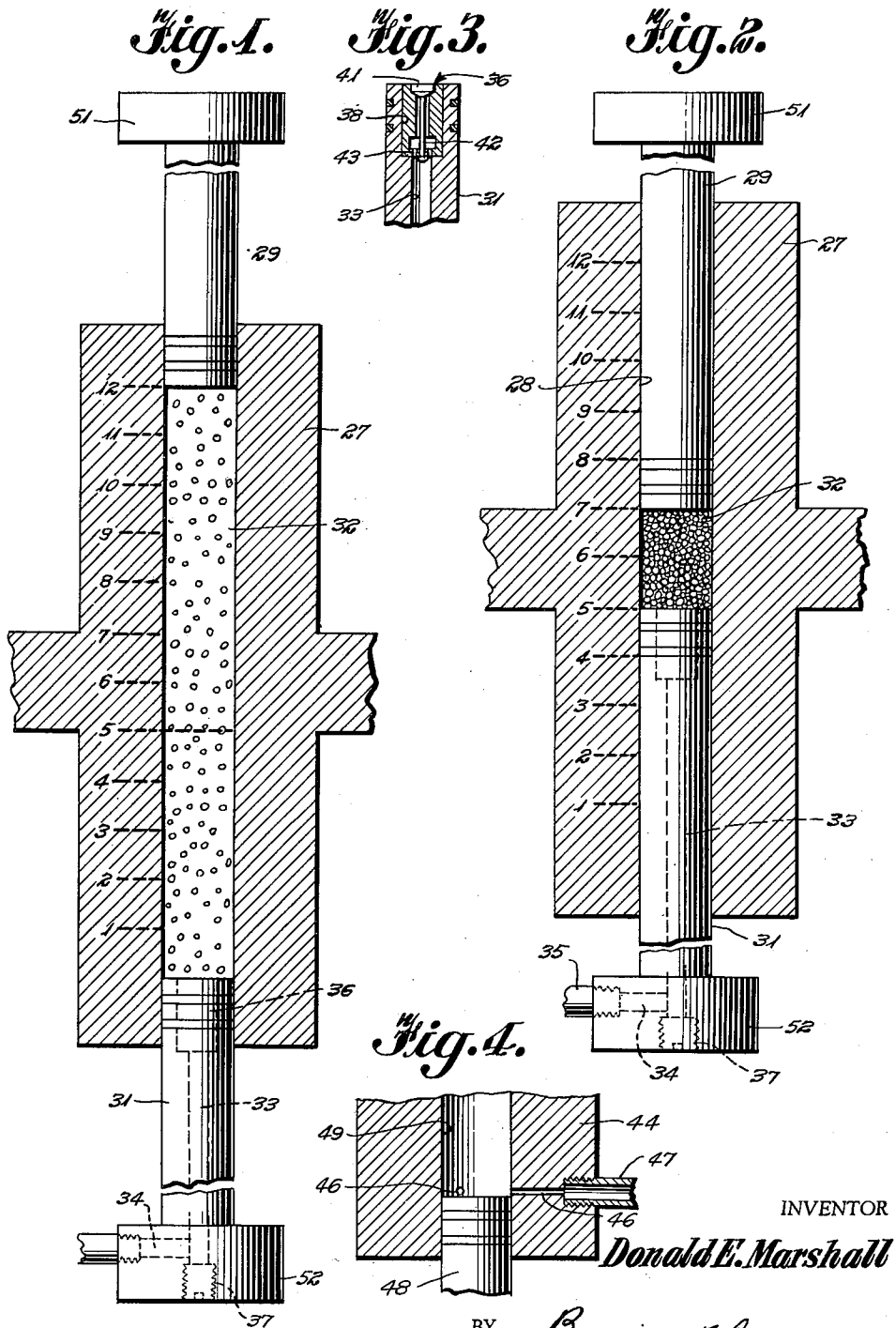
April 29, 1952

D. E. MARSHALL
METHOD OF COMPACTING PULVERIZED MATERIALS
AND PRODUCT RESULTING THEREFROM

2,594,956

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2 SHEETS—SHEET 1



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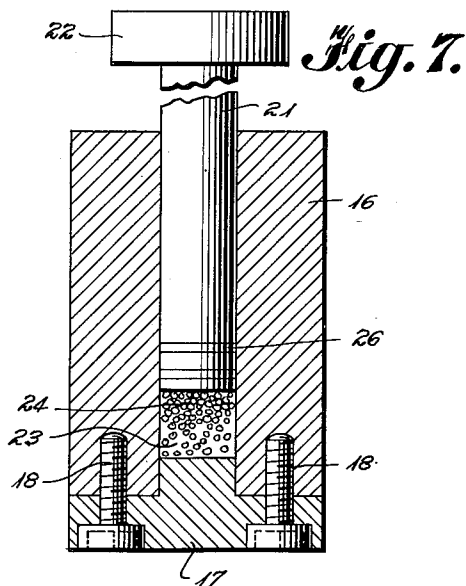
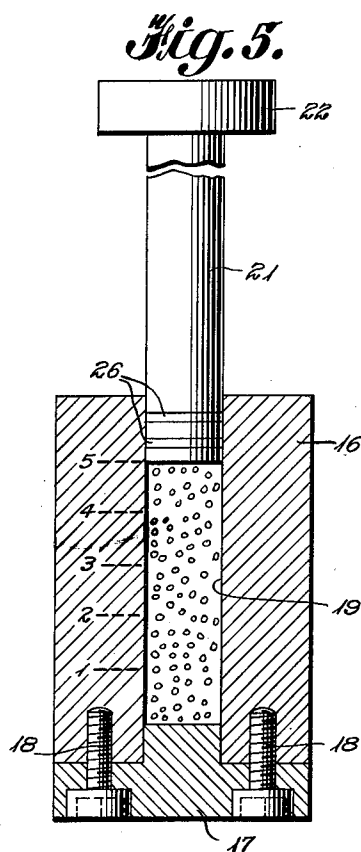
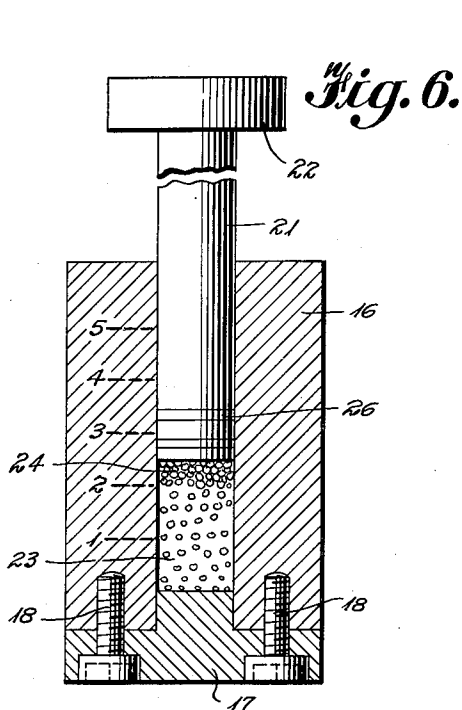
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2 SHEETS—SHEET 2



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METHOD OF COMPACTING PULVERIZED MATERIALS AND PRODUCT RESULTING THEREFROM

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This invention relates to a method for compacting pulverized solid materials, and more particularly, to a method for producing form-retaining bodies of such pulverized solid material in which the particles of the solid material engage each other at spaced points and are partly separated from each other by spaces or voids and the spaces and particles are substantially uniformly arranged in all portions of the bodies so that the bodies have a substantially uniform structure throughout.

In conventional processes of compacting or pressing powdered materials into tablets or cakes, the particles are not uniformly pressed together in all portions of the resulting product. Instead, the portions of the final product adjacent the pressure plunger usually employed are more closely pressed together so that the voids or spaces between the particles in such portions are substantially smaller than in other portions of the compressed product. This, of course, means that if the less closely compacted portions of the product are sufficiently compacted to develop the desired mechanical strength, other portions of the product are over-compressed. The result is that a non-uniform structure is developed and in portions of the product, the particles may even be wedged together and deformed to substantially fill all of the pores or spaces while in other portions, the particles are relatively loosely compacted to leave larger and more numerous spaces between the particles.

In accordance with the present invention, I have developed a process by which solid pulverized material may be compressed into form-retaining bodies which have a substantially uniform structure throughout. That is to say, all portions of the products are subjected to substantially uniform pressure so that the arrangement of particles and spaces is substantially uniform throughout the compressed product. Form-retaining compressed bodies can be produced in which the particles adhere to each other at spaced points and are uniformly partly separated from each other so that the product can readily be crushed into its original pulverized form by slight rubbing pressure, such as crushing between the fingers. This has not been possible by prior compacting processes since portions of the compressed products are over-compacted so as to break into gritty chunks even though other portions may be readily reduced to their original pulverized form, as above discussed.

The results of the present invention are ac-

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complished by employing uniformly pulverized materials and suspending the particles of a predetermined charge of the pulverized material in a gas in a die chamber having an internal volume substantially greater than the normal volume of the charge of pulverized material and then while the particles are suspended in the gas so as to be substantially all separated from each other by such gas, suddenly applying pressure to the suspension to reduce its volume to a volume substantially less than the normal volume of the charge. The pressure may be applied through movable walls of the die chamber to reduce the internal volume of the die chamber. By the "normal volume of the charge" is meant the volume of the charge when the particles are loosely in contact with each other, i. e., the volume the charge would have when in an uncompact state in an open container.

Under the conditions of compacting above described, the gas between the particles resists being driven from between the particles so as to maintain the particles substantially uniformly distributed in the die chamber even when sufficient mechanical pressure is applied to reduce the volume of the charge to such an extent that the particles adhere together. The gas is progressively compressed in the interstices between the particles and tends to produce a substantially uniform distribution of voids with the particles in contact with each other at spaced points. A substantially uniform porous product is thereby produced and upon release of the mechanical pressure, the gas under pressure escapes from the pressed product without disruption thereof leaving a product which is substantially uniform in structure throughout. With such a uniform structure it has been found that sufficient adherence between the particles can be secured to produce compressed bodies which will retain their shaped form under ordinary handling but which can be readily disintegrated into their original pulverized form with slight pressure. The problem here is not so much one of "binders" as it is mechanical interlocking of particle contact areas; in fact, the material must be free enough of moisture, low-melting point solids, or sticky, oily ingredients, to prevent the permanent welding together of these particles under the mechanical and gas pressure, to which they are subjected. Suitable additives, made up of dry, sharp-cornered particles which will interlock particle to particle at the corners to aid in forming a minute honeycomb structure around minute interstitial gas cells are sometimes ad-

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vantageous and may be employed. Sufficient mechanical pressure can, however, be employed to produce formed bodies of higher mechanical strength and if desired, such bodies, if of fusible material, can be sintered to produce substantially uniform structures having even higher mechanical strength. The uniformity of structure discussed above is secured even if binders such as above referred to are employed so long as the powdered material is capable of being substantially uniformly dispersed in a gas.

By employing extremely finely pulverized material, for example materials having substantially uniformly sized particles of the order of 1 or 2 microns in diameter, products having a characteristic soft feel which can be described as creamy may be produced, and which are particularly useful for many purposes, for example in preparations such as face powders, dentifrices, shaving soaps, etc. Methods of producing such finely divided uniformly sized powdered products of substantially any type of material have been developed by me and are disclosed, for example, in my applications Serial No. 32,189, filed June 10, 1948, and Serial No. 654,996, filed March 16, 1946, now Patent No. 2,561,394. For other products not requiring the soft feel referred to above the particle size of the pulverized material may be larger but best results are obtained when substantially all of the particles are very nearly the same size. Thus, for some types of soap or other detergent bars the particle size may be of the order of 6 microns in diameter and for other products such as flour or readily disintegrated powdered soap compacts, the particle size may be very much larger.

The development of a substantial gas pressure in the die chamber prior to compressing the powdered material, i. e., before the volume of the die chamber has been reduced to a volume less than the normal volume of the powdered material, is of assistance in producing the uniform structure above discussed. Thus, the pressure can be increased in the die chamber substantially above atmospheric pressure by introducing a stream of gas under pressure into the die chamber before the suddenly applied compressing force is initiated. Such a stream of gas may be employed to disperse the particles of the charge throughout the volume of gas in the die chamber or may even be employed to introduce the charge of powder in suspension in the gas. The greater the gas pressure developed in the chamber prior to the compressing operation and the greater the resulting gas pressure during the final compression of the powdered material as well as the greater the speed of the plungers all within practical limits, the more uniform the structure of the resulting product. That is to say, the greater the gas pressure, i. e., the amount of gas between the particles, the more tenaciously the gas remains in position to prevent non-uniform pressing together of the particles and the greater the speed of the plungers, the less time available for the gas to escape from between the particles.

The invention is applicable to the preparation of aerated cakes or compacts of dry powders of a large number of materials such as detergents, chemicals, foods, flour, medicines, etc., which, although appearing to be in a caked condition, can be crushed or abraded so as to disintegrate into a pulverized material again. These compacts may be in the form of rectangular bars or cubes, cylindrical cartridges, or thin discs, sticks or cakes having markings so that measured portions

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can be readily broken off for use; or individually wrapped portions which can be crushed before unwrapping. The size of the compact can range from the mouth-tablet size to 25-pound briquettes of cake flour, compacted for economical packaging.

Another use of this invention relates to making bar soaps, or bars of synthetic detergents, from ingredients which are impossible to integrate by ordinary extrusion methods, because of the abrasive action of the ingredients on the extrusion machinery, or the poor welding properties of the ingredients, which can be inert dry builders plus extremely low-moisture (anhydrous) soap powders, etc.

Such ingredients in the present invention are first blended as substantially uniformly sized powdered particles, and then intensely compacted in a casting die to make a form-retaining, uniformly aerated mass or compact of the desired density, and then this compact is made serviceable as a toilet bar by sintering the lowest-melting-point ingredient to weld the particles together more securely.

The sintering may be done coincident with compacting, and result only from the effect of compacting pressure in lowering the melting-point of a binding ingredient, accompanied by the heat generated in compressing the gases which are present; or the sintering may be done as a tempering step following the compacting step, by the use of properly regulated ovens or by employing electro-static heating, so that the bar is sintered uniformly throughout its body.

It is therefore an object of the present invention to provide an improved process for compacting pulverized solid material to produce form-retaining porous bodies having a substantially uniform structure throughout.

Another object of the invention is to provide a process for producing form-retaining bodies of compacted powdered material having a substantially uniform porous structure throughout in which process a substantially uniform suspension of a powder in a gas is subjected to mechanical pressure to bring the particles into contact and cause them to adhere together.

Another object of the invention is to provide a process for producing form-retaining bodies of compacted powdered material in which a substantially uniform suspension of powdered material in a gas under pressure is suddenly further compressed to bring the particles of powdered material into contact and form compacted bodies having a substantially uniform structure throughout.

A further object of the invention is to provide an improved product made of compressed pulverulent material, which product is form-retaining and has a substantially uniform porous structure throughout.

A still further object of the invention is to provide a porous form-retaining product made of compressed finely divided powdered material which has a substantially uniform structure and which may be readily reduced to its original finely divided form.

Other objects and advantages will appear in the following detailed description thereof given in connection with the attached drawings, in which:

Fig. 1 is a diagrammatic vertical cross-section through a die structure in accordance with the present invention showing compressing plungers in their extended or greatest volume position;

Fig. 2 is a view similar to Fig. 1 showing the plungers in their compression or minimum volume position;

Fig. 3 is a fragmentary vertical cross-section through the inner end of a suitable die plunger;

Fig. 4 is a fragmentary vertical cross-section through a modified die structure showing an alternative manner of introducing gas;

Fig. 5 is a diagrammatic vertical cross-section through a die structure operating in accordance with the prior art showing a die plunger in its maximum volume position;

Fig. 6 is a view similar to Fig. 5 showing the die plunger in an intermediate position; and

Fig. 7 is a view similar to Fig. 5 showing the die plunger in its minimum volume position.

Referring more particularly to the drawings, reference is first made to Figs. 5 to 7, inclusive, as an aid to understanding the present invention. In Fig. 5, the die structure is provided with a die-shroud 16 having one end closed by a removable head 17 secured to the die-shroud 16 in any suitable manner such as by cap screws 18. As shown, the die-shroud 16 provides an interior substantially cylindrical die chamber 19 into which a compression plunger 21 may be forced by any suitable means acting upon the head 22 of the die plunger. In conventional processes for pressing powder into form-retaining bodies, a charge of powdered material 23 having a normal volume substantially filling the chamber is introduced into the chamber 19. By normal volume is meant the volume of the powder when the particles thereof are loosely in contact with each other, although in Fig. 5 the various particles of the charge are represented as being separated from each other for clarity of illustration. In such prior art operations the plunger 21 is driven into the die chamber 19 to compress the powder and Fig. 6 illustrates diagrammatically approximately what happens in the die chamber at an intermediate position of the compression plunger 21. When pressure is initially applied through the plunger 21 a portion of this pressure is exerted laterally against the walls of the pressure chamber. This may be considered to be due to wedging of certain of the particles in the upper portion of the charge between other particles and the result is the development of a high frictional force adjacent the walls of the die chamber resisting movement of the powder. This produces greater compression of the particles at 24 adjacent the plunger 21 and this condition persists until the powder is compressed into its final form illustrated in Fig. 7 in which the upper portion 24 of the charge 23 is much more highly compressed than the lower portion. If the lower portion of the charge is sufficiently compressed to have the desired mechanical strength the upper portion of the charge is over-compressed. For example, if the lower portion of the compressed body can be again pulverized into its original form by slight pressure, the upper portion of the compressed body will be difficult to disintegrate and will break into chunks rather than into its original powdered form under the same pressure.

Another reason for the failure to produce a uniform structure in processes such as illustrated with respect to Figs. 5 to 7, inclusive, is the low gas pressure which is developed during the compression of the powder even if the die chamber 19 is gas-tight and the plunger 21 should be provided with piston rings or other sealing means 26. For example, assume that the original nor-

mal or apparent volume of powdered material represented by the numeral 5 in Fig. 5 is 5 times its actual volume, i. e., its volume when no voids exist in a compressed structure. In this case the volume of gas in the die chamber is four times the actual volume of the powdered material at atmospheric pressure which may be assumed to be 15 lbs. per square inch. When the powder has been compressed to one-half of its original volume, as illustrated in Fig. 6, the original four volumes of the gas has been reduced to $1\frac{1}{2}$ volumes and even under these conditions the gas pressure is only 40 lbs. per square inch. This is a small fraction of the mechanical pressure required to compress the powder to one-half its original volume, which mechanical pressure will usually range from 200 lbs. per square inch upward.

In Fig. 7 the powder is shown as having been compressed to approximately one-fourth its original volume such that the voids in the compressed product are 20% of the final volume. This is a greater compression than is ordinarily employed and will ordinarily require pressure in excess of 1000 lbs. per square inch or more. In this case the original four volumes of gas has been reduced to $\frac{1}{4}$ volume or to $\frac{1}{16}$ of the four volumes. However, the gas pressure is only 240 lbs. per square inch. Again the gas pressure is a relatively small part of the total pressure required to compress the powder. The important factor, however, is that the low gas pressure, i. e., the small amount of gas present in conventional compacting processes, allows the gas to be easily driven out of a portion of the charge during the initial stages of compression of the powder and trapped in another portion thereof so as to produce a non-uniform product.

In accordance with the present invention, as illustrated in connection with Figs. 1 to 4, inclusive, a die-shroud 27 is employed having a die chamber 28 providing an internal volume which is much greater than the normal volume of the powdered material. In the preferred die structure, the die chamber 28 extends all the way through the die shroud 27 and a pair of pressure plungers 29 and 31 extending into opposite ends of the die chamber 28 are provided. In Fig. 1 the internal volume of the die chamber 28 is represented as being 12 times the actual compressed volume of the powdered material, and again the normal volume of the charge may be assumed to be five times the actual volume of the solid material as represented by the dotted line opposite the numeral 5 in Fig. 1. In Fig. 1 the particles of the charge 32 are represented as being distributed throughout the volume of the die chamber 28 and this distribution or suspension of the particles in the gas can be accomplished by jetting air or other gas into the bottom of the chamber after the charge has been introduced thereinto. A suitable structure for this apparatus is illustrated in Figs. 1, 2 and 3. That is to say, the lower pressure plunger 31 may be provided with suitable intersecting ducts 33 and 34 providing a conduit from a pipe 35 to a suitable valve such as a check valve structure 36 illustrated in Fig. 3, the duct 33 having its lower end closed by a plug 37. For example, the duct 33 in the plunger 31 may terminate at its upper end in an enlarged recess 38 for receiving a valve casing 39 in which is positioned a valve member 41. The valve member 41 may have a stem 42 extending down through a central bore in the valve casing 39 which stem may be riveted

to a perforated plate 43 sliding in an enlarged portion of the central bore in the valve casing 39. The valve casing 39 may have a press fit in the recess 38 in the piston 31 and it will be apparent that introduction of gas under pressure through the duct 33 will open the valve 41 to introduce a jet of gas into the charge 32 in the lower portion of the die chamber 28 to cause particles of the charge to be blown upwardly into the gas in the die chamber to substantially uniformly suspend the particles in such gas. Introduction of the gas through the duct 33 will at the same time increase the gas pressure in the die chamber 28.

If at the time or shortly after the gas is introduced into the die chamber 28 the plungers 29 and 31 are suddenly driven into the die chamber 28, for example to the position illustrated in Fig. 2, the solid particles are compressed into a form-retaining body having a substantially uniform structure throughout. That is to say, the particles at the beginning of the pressure stroke are substantially completely separated from each other by gas and during compression this gas resists being driven from between the particles so that the particles are brought into contact with each other at spaced points. The compressed gas also resists being driven from between the particles and the walls of the chamber so as to minimize sliding friction. The gas under pressure, therefore, remains substantially uniformly distributed throughout the compressed mass so that the spaces between the portions of the particles not in contact remain substantially uniformly distributed throughout the compressed mass. The inertia of the check valve 41 causes closure of this valve during the initial portion of the pressure stroke of the piston 31 and this may be timed so that the valve closes while the gas is still being introduced through the check valve 41. This prevents clogging of the check valve with powdered material since the introduction of the gas prevents any forcing of powdered material into the valve when the valve is open. As soon as the valve closes, a substantially smooth surface is presented to the charge being compressed.

An alternative method of introducing the suspending gas is illustrated in Fig. 4. In Fig. 4 a die shroud 44 may be provided with a plurality of circumferentially spaced apertures 45 communicating with pipes 47 screw-threaded into the exterior wall of the die shroud, the apertures 46 being positioned just slightly above the lowermost position of the lower pressure plunger 48. If a charge has been previously placed in the die chamber 43, introduction of the gas under pressure through the apertures 46 will cause suspension of the powdered particles throughout the die chamber in the same manner as illustrated in Fig. 1. In fact, the entire charge may be introduced through apertures 46 already in suspension in the gas if suitable charge-forming apparatus is employed. Initial movement of the plunger 48 in an upward direction closes the apertures 46 so that escape of gas and powdered material is prevented during the pressure stroke.

It will be apparent that any suitable source of power may be employed to suddenly drive the plungers into the die chambers of Figs. 1 and 2. That is to say, impact or hammer blows from cam or spring driven weights may be applied to the heads 51 and 52 or the plunger may be directly driven from quick acting pneumatic or hydraulic cylinders. In any case the heads 51

ing the plungers into the die chamber. Two or more plungers entering into a single confined space, as illustrated in Figs. 4 and 5, are preferred but it will be apparent that a single plunger of greater length may be employed. It will also be understood that the apparatus will be provided with suitable charge forming devices and with ejecting mechanism for the compressed bodies and that a plurality of dies, for example, mounted on a turret, may be employed. The injection of suspending gas or of gas containing the powdered charge in suspension may be timed with respect to the movement of the plungers, for example, by a cylinder having a piston directly connected to the plunger driving mechanism.

In the apparatus of Figs. 1 to 4, a relatively large volume of gas is compressed even before the volume of the powder is compressed to its assumed normal or apparent volume of five times its actual volume. For example, assume that 500 lbs. per square inch total pressure is required to the compress the gas and charge to twice the actual volume of the powder and that the gas has an initial pressure of 40 lbs. per square inch. The gas is compressed to $\frac{1}{11}$ its original volume to produce a gas pressure of 440 lbs. per square inch. Since the charge is fluidized by gas between the particles during the initial compression and this effect persists even after the particles have made actual contact with each other, the final pressure is substantially uniformly exerted in all directions in the charge. During the small time-elapse under high-speed or impact-type compression, resistance to interstitial gas flowing within the compacted material develops. As a result there are no portions of the compacted material where the interstitial gas has been squeezed out. Under these conditions the remaining 60 lbs. per square inch pressure, attributable to the resistance of the particles to being forced together, is sufficient to produce a form-retaining body from many types of powder, which body may be easily reduced to substantially its original powdered condition with slight mechanical working.

The above is given merely by way of example as the nature of the powdered material to be compacted and the properties desired in the compressed product will vary widely. That is to say, a ratio of the total volume of the die chamber to the apparent volume of the charge required to produce a form-retaining body as well as the initial gas pressure employed will depend upon these factors. The travel of the plunger can be reduced by using a greater gas charge, but tests have shown that the proportions shown are desirable in most cases to offset the plunger leakage when making a well-compacted tablet, such as a tablet to be chewed, or a toilet soap bar to be sintered, which may require a plunger force of over 1,000 lbs. for each square inch of plunger area. However, a die chamber having a volume considerably greater than the normal volume of the powder charge and with a greater plunger travel will with certain types of powdered material render the introduction of additional gas unnecessary. That is to say, the initial movement of the plunger causes turbulence in the die chamber to disperse the particles in the gas in the chamber and the enlarged volume of the die chamber provides for building up the desired gas pressure. In this specification the gas pressures referred to are absolute pressures.

The application of the aerated compacting

process of the present invention to the manufacture of toilet articles introduces a new form of product which differs from any product heretofore known. This invention makes a compact which, when crushed in the hair as a shampoo powder, will adhere to the head during the application of water, in a manner similar to the adherence of a cream shampoo. This new characteristic, inherent in this new compact, is derived from the use of extremely fine pulverized ingredients (approximately 2 microns or less being the particle diameter) plus the aeration, which causes the fine particles to "spread" more like a cream than a powder.

Likewise, a shaving powder preparation reduced to this fine pulverization, and compacted with aeration, will make a tablet which, if pressed on the chin below the lips, will adhere and when worked with the shaving brush will develop a lather as satisfactory as a cream, but with a much easier to use package than a collapsible tube; and in a measured form, in place of a squeezed-out, unmeasured amount of cream.

The spreadable consistency is not dependent upon a waxy ingredient or oily binder, but is the result of providing an extent of pulverization and of aeration and an extent of mechanical compacting which makes a compact that will crush in this new and desirable manner. If the ultra-fine pulverized ingredients needed for the shampoo or shave powder tablet were compacted without aeration, the resulting cake would be so hard, and the particles so amalgamated as not to crush into a spreadable material.

The exact formulation of the above products is not a part of this invention and therefore only general definitions will be given to clarify the nature of the new products, which do not include substantial amounts of water or other cream-forming ingredients, such as glycerin, mineral oil, triethanolamine, Irish moss gel, etc.

A suitable shampoo powder formulation may include the dehydrated active ingredient of a mild wetting agent such as sodium lauric monoglyceride sulfate, combined with sufficient inert builder such as magnesium carbonate, to absorb the desired perfume oils so that the blend of wetting agent, builder and essential oil will pulverize satisfactorily in an apparatus such as is defined in my application Serial No. 32,139, supra. This blend pulverized to an average particle size of approximately 2 microns and compacted with an aeration of 25 to 50% at a mechanical pressure of 200 to 500 lbs. per square inch of plunger area.

The degree of aeration and mechanical pressure may be determined by pilot tests since such factors as the temperature of the charge under compression must be regulated to avoid agglomerations which do not crush and spread satisfactorily. Also, small amounts of hair conditioning organic resin-like materials, which may be an added ingredient, can have a very large effect on the compacting conditions. Nevertheless, a combination can be found by varying the builder, the temperature of pulverization, etc., which will make it possible to prepare an ultra-fine powder and once the powder is prepared, the conditions of compacting may be adjustable to gain a crushable compact which crushes and spreads satisfactorily.

A suitable shaving powder formulation may be any stearic acid, coconut oil, potassium soap combination which makes the consistency of lather desired, plus the desired perfume, and a small amount of dry builder, such as titanium dioxide, to absorb the essential oil. The soap

portion should be spray-dried down to as near anhydrous as possible, for example, below 2% moisture. This blend may then be pulverized down to an average particle size of approximately 2 microns in diameter. The compacting should be adjusted to yield a tablet which is well aerated, for example, approximately 25% voids and integrated with approximately 200 lbs. to 500 lbs. per square inch of plunger force. The finished tablet should crumble and spread easily, and furnish the right amount of lather for a good shave when worked up with a wet shaving brush.

The present invention may also be employed to produce an improved type of dentifrice. Tooth powder tablets may be made which contain such finely pulverized abrasive ingredients, and in such uniformly aerated compacted form, that for the first time such a preparation, with its proper flavoring and foaming constituents, will seem to dissolve in the mouth like an after-dinner mint with no sensation of grittiness or of medicine-like powder.

There has been much attention given in the past to the preparation of dentifrices which can be conveniently applied to a toothbrush. Among these are tubes providing ribbon-shaped creams, powder cans with spouts correctly sized for the brush and liquid preparations given a gel consistency so as to cling to the brush. The present invention enables the production of a dentifrice suitable for placing directly in the mouth, and of a nature suitable to cleanse the mouth with or without a small amount of water, and with or without a tooth-brush. The flavoring ingredient and wetting agent may be regulated so as to stimulate the flow of saliva and reduce its surface tension sufficiently to form a solution which suspends the extremely fine inert builders or abrasives. This invention has resulted in the discovery that fine pulverization plus aeration will yield a tablet which can be used in this manner, that is, placed in the mouth and crushed with the teeth to develop a cleansing mixture which is palatable.

The tooth and mouth senses will detect the grittiness of abrasives if pulverized only to the 6 micron size, particularly when such a tablet is bitten into. So the degree of pulverization is important for palatability considerations. Furthermore, if such ultra-fine loose powder is placed in the mouth, the drying action all over the mouth is so intense as to gag the user. However, by preparing this same powder into a properly compacted aerated tablet, this distasteful effect is absent. The ultra-fine pulverization of the abrasive also has merit in the polishing action on the tooth enamel. The finer the abrasive, the higher the polish and the less damage is done to the teeth. If the extremely fine material contemplated by the present invention were compacted by the methods of the prior art discussed above, a non-uniform structure would result in which at least a portion of the pellet or tablet would be pressed into a hard material having a gritty texture. It was not until it was discovered that a hard and gritty material such as abrasive could be reduced to such a fine state of division that it could not be detected as gritty material in the mouth, and it was further discovered that such a fine material could be compacted into a form-retaining body without again producing gritty aggregates, that useful tooth powder pellets or tablets could be produced. The range of particle size for the abrasive or other gritty material is from about 1 to 4 microns, although softer materials, such as the foaming agent in the

blend, may have a particle size as great as 6 microns.

A tooth powder formulation suitable for this technique may consist of from 4% to 5% foaming agent such as sodium lauryl sulfate in anhydrous powder form blended with suitable powdered abrasives, such as mixtures of chalk, calcium carbonate, magnesium oxide, magnesium hydroxide, titanium dioxide, etc., plus flavoring oils, such as peppermint oil. This blend may be pulverized down to an average particle size of approximately 1 micron and then compacted with a good aeration of 30 to 40% at compacting forces of 100 lbs. to 300 lbs. per square inch of plunger force to gain the texture and chewing properties desired. The new decay deterrents, such as fluorine and ammoniated phosphates with urea, etc., can be added to this formulation. The fact that ultra-fine pulverization is accomplished plus a procedure that holds the dentifrice in the mouth and thus results in good distribution of the medication make this dentifrice form ideal for the incorporation and use of decay-deterrents.

The application of this invention to the manufacture of toilet bars, either from soap or synthetic base materials, is important in several respects if full consideration is given to the bar-preparation technology and its limitations as practiced heretofore. The present invention may be employed to make aerated bars of extremely low-moisture content, which heretofore has been considered below the practical range, that is, well below 12% moisture. Furthermore, this low soap can first be converted to the so-called transparent phase, with ultra-fine texture and other desirable qualities, before or during pulverization, so that when integrated at room temperature into an aerated compact, these refined qualities are preserved and the soap does not revert to coarser crystalline structures, which would result if excessively heated.

Special bar structures are possible by employing the present invention. For example, the cushion or border of the bar may be aerated sufficiently to cause the bar to float but the center panel may be compacted additionally in a further pressing operation so that it is de-aerated and is translucent so as to show that the bar has been refined to the ultra-microcrystalline state.

Synthetic detergent bars may be produced by this invention to eliminate the discoloration which results from the abrasive character of this material wearing off the milling equipment and plodders used for conventional processing of milled and extruded bars. Synthetic detergent ingredients plus the adulterating sodium sulfate salts which are produced in the neutralizing process are pulverized, together with suitable inert builders such as pumice, chalk, titanium dioxide, etc., until a particle size of approximately 4 microns is reached. Such a blend can be formed into an aerated compact without picking up discolorations due to abrasion, or without developing over-sized salt crystals. The bar texture depends more upon how uniformly pulverized and uniformly bonded the ultra-fine particles are than it does upon the nature of the particles, i. e., whether the particles are of inert insoluble builders or soluble soap and synthetic detergent crystals. The body structure of the bar may be made from inert powdered builders if the detergent active ingredient is too soluble to make a durable body. This detergent active ingredient may be either an over-soluble synthetic or an

over-soluble soap. In the case of the latter, the body may be developed from less soluble stearic acid soaps in place of, or in addition to, the inert builder powder. However, the principle is the same, as will be explained below.

The body structure of the bar may be largely independent of the active detergent so far as its degree of disintegration in use is concerned. For example, a bar can be compacted from an ultra-fine blend of chalk, calcium carbonate and titanium dioxide, with only 5% of synthetic detergent such as sodium lauryl sulfate, which will feel smooth and will wash away in use at the rate desired. The size of the inert particle determines the smoothness of the bar while the degree of aerated, mechanical compacting largely determines its disintegration in use. If such a bar as just described is prepared from 2 micron material, and properly compacted, it will wear away at the desired rate and have the desired smooth texture and feel no denser than a milled bar, when lifted. The only deficiency is lack of adequate lather because 5% is not enough active detergent. A very satisfactory bar can be prepared as above, however, if the active detergent ingredient is increased to 60% and the body builders reduced to 40% by weight. The increase in active detergent seems to moderate the texture so that 4 micron to 6 micron sized builder powders are as fine a pulverization as is needed. The aeration required is 15% to 20% and the compacting force can be from 500 lbs. to 2,000 lbs. per square inch of plunger force depending upon the degree of sintering which takes place in the compacting operation due to the temperature of the charge of powder and gas being compressed plus the lowering of the melting point of the active detergent, due to pressure. This sintering of bars made from powders, either during the compacting or as a tempering step following the compacting, is necessary to get a durable bar all the way through its use. The degree of sintering needed depends upon the ingredients and just how these ingredients disintegrate as the bar is wetted in use. For example, a bar in which the active detergent is a pulverized soap in the transparent state will require little or no sintering because this material makes a cold weld every time it is wetted, being in the jelly or ultra-microcrystalline state.

The above example of a bar made from inert pulverized builder and synthetic detergent has a body structure that is largely independent of the solubility of the active detergent. Its disintegration in use is regulated more by the degree of aeration and the degree of bond between each particle of inert material. The amount and character of active detergent affects this bond but their effect can be compensated for by varying the conditions of compacting.

It has been customary to blend soaps of different titre stocks to get the body and solubility desired in a bar. In the case of soaps, such blends have given the body structures desired, especially when the degree of moisture and degree of milling also furnished additional controls over texture. With anhydrous soaps this becomes more difficult. The present invention may, however, be used to produce low-moisture soap aerated bars in much the same manner as described above. That is, a body structure is built with finely pulverized hard stearic acid soaps and the softer soap quick-lather ingredients of the formula are made into powders and blended uniformly before aerated compacting. In such a technique the disintegra-

tion in use coefficient can be regulated largely by the harder soap portion and the degree of aeration and compacting given to it.

There are many important applications of this regulated compacting and aerating process of the present invention for preparing measured portions of material and packaging these compact, aerated portions into packages reduced in volume approximately 50% without agglomeration difficulties.

Ground coffee, prepared in compacts of the right size for a coffee-potful, can be individually wrapped and then contained in a can under vacuum, with a saving of one-third to one-half the tin needed otherwise, and with the contents protected after the can is once opened. These wrapped portions are designed to be squeezed back into granulated form as the coffee pot is charged.

Granulated soaps and synthetic detergents prepared in crushable cubes of the right size for a dishpanful of dishes will prevent waste. Heretofore such materials have been bulked up, by making the product of extremely light density, so its use could be stretched out. By compacting a concentrated, water-free, builder-free, excess air-free detergent powder, the savings of packaging materials and storage space, amounting to one-half of what these items cost at present, are possible without the consumer being apt to waste such a concentrated soap. This is because it is in measured portions to be counted out, instead of being dumped into the washing machine or tub.

It is known that fine laundry milled soap flakes are first baled into a compact mass and then the bale is placed in the carton to insure bulging fullness when the carton is opened by the consumer. The present discovery makes possible the molding of a form-retaining cake of powdered detergents which can be placed in the carton to give compactness and fullness of the carton not possible in any other way. Such a material is compacted so as to re-pulverize if the carton is squeezed or if individual portions are crushed.

At the proper temperature and dryness, an extremely fine wheat flour can be compacted into aerated, form-retaining measured units which will sift in the kitchen equipment as satisfactorily as loose flour and be easier to use. This is also true of cocoa, baking powder, dry yeasts, sugar, etc., all of which can be in pre-measured portions.

Candies with a new delightful creamy solubility can be made from compacted, extremely fine pulverized sugars and chocolate, making possible an entirely new confection product and technique. The features are the elimination of heat, both during the pulverization and compacting so that creamy, ultra-fine materials are not made coarse and sugary by the heat and aeration is added to give the proper chewing texture.

Insecticides which are carefully pulverized for dusting crops may be packaged with large paper savings by the process of the present invention. Many other chemicals lend themselves to this method, such as photographic powders, and particularly those needing protection from moisture after a large package has once been opened. In accordance with the present invention, individually-wrapped "crush-cubes" may be provided.

I claim:

1. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an

internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, dispersing the particles of said charge in a gas in said chamber, and while said particles are thus dispersed suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

2. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, directing a jet of gas into the lower portion of said chamber to disperse the particles of said charge so that said particles are suspended in and separated from each other by the gas in said chamber, and while said particles are thus suspended suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

3. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, said charge being introduced into said chamber in suspension in a stream of gas so that said particles are suspended in and separated from each other by the gas in said chamber, and while said particles are thus suspended suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

4. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, introducing a gas under pressure into said chamber to increase the gas pressure therein above atmospheric pressure, dispersing the particles of said charge in the gas in said chamber, and while said particles are thus dispersed suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

5. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, directing a jet of gas under pressure into the lower portion of said chamber to increase the gas pressure in said chamber above atmospheric pressure and disperse the particles of said charge so that said particles are suspended in and separated from each other by the gas in said chamber, and while said particles are thus suspended suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

6. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an internal volume substantially greater than the

normal volume of said charge when the particles of said charge are loosely in contact with each other, said charge being introduced into said chamber in suspension in a stream of gas under pressure so that said particles are suspended in and separated from each other by the gas in said chamber and the pressure of the gas in said chamber is increased above atmospheric pressure, and while said particles are thus suspended suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

7. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, introducing a stream of gas under pressure into said chamber to increase the gas pressure therein above atmospheric pressure, and suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

8. The method of compacting pulverized solid material, which comprises, introducing a charge of said material into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, dispersing the particles of said charge in a gas in said chamber, and while said particles are thus dispersed suddenly decreasing the internal volume of said chamber to less than said normal volume by mechanical pressure exerted through movable walls of said chamber to simultaneously compress said gas and press said particles together.

9. The method of compacting pulverized solid material, which comprises introducing a charge of said material into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, dispersing the particles of said charge in a gas in said chamber, and while said particles are thus dispersed suddenly decreasing the internal volume of said chamber to less than said normal volume by mechanical pressure applied by driving into said chamber opposed plungers fitting said chamber to simultaneously compress said gas and press said particles together.

10. The method of compacting pulverized solid material, which comprises, introducing a charge of said material in which the particles are substantially of the same size and have a diameter between 1 and 6 microns into a die chamber having an internal volume substantially greater than the normal volume of said charge when the particles of said charge are loosely in contact with each other, dispersing the particles of said charge in a gas in said chamber, and while said particles are thus dispersed suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

11. The method of compacting pulverized solid material, which comprises, introducing a charge of said material in which the particles are substantially of the same size and have a diameter between 1 and 6 microns into a die chamber having an internal volume substantially greater than the normal volume of said charge when the par-

ticles of said charge are loosely in contact with each other, introducing a stream of gas under pressure into said chamber to increase the gas pressure therein above atmospheric pressure, and suddenly decreasing the internal volume of said chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

12. The method of compacting pulverized solid material to produce form-retaining bodies in which the particles of solid material engage each other at spaced points and are partly separated from each other by spaces and said spaces and particles are substantially uniformly arranged in all portions of said body so that said bodies have a substantially uniform structure throughout, which method comprises, introducing a charge of said pulverized solid material into a die chamber having an internal volume substantially greater than the normal volume occupied by said charge when said particles are loosely in contact with each other, introducing a gas into said chamber to increase the gas pressure therein above atmospheric pressure, dispersing the particles of said charge in the gas in said chamber so that said particles are suspended in said gas and are substantially all separated from each other by said gas, and while said particles are thus suspended suddenly decreasing the internal volume of said die chamber to a volume substantially less than said normal volume to compress said gas and just sufficient to press said particles together to produce form-retaining bodies which may be readily disintegrated into substantially the original pulverized solid material.

13. The method of compacting pulverized solid material to produce form-retaining bodies in which the particles of solid material engage each other at spaced points and are partly separated from each other by spaces and said spaces and particles are substantially uniformly arranged in all portions of said body so that said bodies have a substantially uniform structure throughout, which method comprises, introducing a charge of said pulverized solid material into a die chamber having an internal volume substantially greater than the normal volume occupied by said charge when said particles are loosely in contact with each other, introducing a gas into said chamber to increase the gas pressure therein above atmospheric pressure, dispersing the particles of said charge in the gas in said chamber so that said particles are suspended in said gas and are substantially all separated from each other by said gas, and while said particles are thus suspended suddenly applying sufficient mechanical pressure through a movable wall of said die chamber to decrease the internal volume of said die chamber to a volume substantially less than said normal volume so as to compress said gas and just sufficient to press said particles together to produce form-retaining bodies which may be readily disintegrated into substantially the original pulverized material.

14. A method of producing aerated shaped bodies of detergent material, which comprises: reducing the selected detergent material to a pulverized state having a solid particle size on the order of less than 6 microns; charging a predetermined amount of the pulverized detergent material into a die chamber along with sufficient air to thoroughly fluidize the charge; and suddenly decreasing the volume of the die chamber with a speed which develops a high interstitial

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resistance to the flow of the fluidizing air within the charge and precludes any appreciable loss of fluidizing air from the die chamber, to thereby compress the air and compact the pulverized detergent material into a uniformly aerated body.

15. An aerated shaped detergent body comprising: pulverized detergent material having a solid particle size on the order of less than 6 microns compacted into cohesion, but with air filled interstitial pores substantially uniformly distributed throughout the body, said body being obtained by charging a predetermined amount of the pulverized detergent material into a die chamber along with sufficient air to thoroughly fluidize the charge, and suddenly decreasing the volume of the die chamber with a speed which develops a high interstitial resistance to the flow of the fluidizing air within the charge and precludes any appreciable loss of fluidizing air from the die chamber.

16. The product of claim 15 but wherein the pulverized detergent material is composed of hard soap of less than 11% moisture and a soft over-soluble detergent well blended and interspersed with one another so that the low moisture fraction protects the softer fraction and governs the hardness of the body.

17. The product of claim 15 but wherein the pulverized detergent material is composed of hard soap of less than 11% moisture and a soft over-soluble detergent plus a pulverized inert builder having a particle size smaller than approximately 4 microns well blended and interspersed with one another so that the hard low moisture fraction together with the inert builder protects the softer fraction and governs the hardness of the body.

18. A form-retaining shaped detergent body comprising: dry solid particles of pulverized detergent material compacted into cohesion, but with air-filled interstitial pores substantially uniformly distributed throughout the body, the cohesion between the particles of pulverized detergent material being sufficient to retain the shape of the body during normal handling while enabling the body to be intentionally readily disintegrated to its original pulverized state by the application of mechanical force thereon, said

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body being obtained by dispersing a charge of said particles in a gas in a die chamber which has an internal volume substantially greater than the normal volume of said charge when the particles thereof are loosely in contact with each other, and while said particles are thus dispersed suddenly decreasing the internal volume of the die chamber to less than said normal volume to simultaneously compress said gas and press said particles together.

19. The form retaining shaped detergent body of claim 18 in the form of a crushable dentifrice tablet containing particles of abrasive material having a particle size ranging between 1 and 4 microns.

20. The product of claim 15 but wherein the pulverized detergent material is composed of hard soap of less than 11% moisture.

21. The aerated shaped detergent body of claim 15 in the form of a dentifrice tablet but wherein the pulverized detergent material contains an abrasive having a particle size less than 6 microns.

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