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PROCESS FOR TREATING SPRAYED DRIED ORGANIC DETERGENTS

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This invention relates to methods for treating materials, such as soaps and synthetic detergents, and to products produced thereby. In particular the present invention relates to processes for increasing the apparent specific gravity and increasing the uniformity in size of particles, and decreasing the amount of fines or dust, in spray dried detergents.

Detergents which have been spray dried are generally relatively porous by reason of shrinkage of the material or the evaporation of moisture therefrom. As a result, they frequently are hollow and may be spongy, or made up of a lattice of material. Because of the character of the particles, they are relatively light in weight as compared with their bulk and have a low apparent specific gravity. Packages and other containers in which such products are shipped, stored or sold, therefore, must be relatively large in order to hold a given weight of the product. At the same time, the porous and fragile nature of the particles renders them weak and friable, so that they tend to form dust when handled and packaged or during shipment. Furthermore, the low specific gravity and small size of the particles appear to promote lumping of the material in hot water.

In copending application Serial No. 507,185, one method for increasing the apparent specific gravity and reducing the dusting and lumping of particulated detergents is described, wherein particles of materials are sprayed with water or a solution of a coating composition, or other material, for modifying the characteristics of the particles. The particles so moistened are tumbled or otherwise agitated, so that they not only are thoroughly coated but are compacted and made more dense, whereby the specific gravity of the particles is increased. However, there is always the possibility that some particles of the material thus treated will be moistened or coated to excess while others are insufficiently treated. Under such conditions, the excessively moistened particles tend to agglomerate and compact into large groups or lumps, while other particles remain untreated or may break down into fines or dust, so that the finished product lacks the desired uniformity. Therefore, such processes often require a degree of control which is difficult to maintain in the usual manufacturing operations.

It has now been discovered that these objections and limitations to prior methods can be overcome, and that spray dried particles of synthetic detergents, as well as spray dried soaps, can be compacted and increased in density, and the formation of dust therefrom inhibited, by subjecting such materials to the action of steam alone or to the action of steam in combination with water or a solution of a coating or modifying agent, while tumbling or otherwise agitating the material. Such treatment overcomes the danger of uneven or excessive moistening of the particles and generally results in a more uniform product. The steaming of the material also is found to speed up the operation so that the desired increase in density and uniformity of the product, and the decrease in dust, can be effected in a relatively short length of time.

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While the invention is not limited to any particular theory of operation, it seems probable that the steam employed condenses most rapidly on the coolest particles of material treated, and at the same time raises the temperature of the particles. In this way, the moistening of the particles is selective, and, as the operation continues, those particles which are untreated and insufficiently moistened, and therefore, cooler, serve to condense the steam to a greater extent than the warmer, previously moistened particles. Uniformity of treatment is thus assured, and the rate of condensation of moisture on the particles decreases as the temperature of the particles increases. Thus the operation is substantially automatic and over-wetting of the particles is easily avoided, whereas drying of the product is facilitated because none of the particles are moistened to excess. Moreover, the particles thus already moistened are conditioned so as to render them more receptive to a liquid treating, plasticizing or modifying agent when employed.

The present invention also renders it possible to produce particulated detergents having the desired high density while possessing a low moisture content and a high percentage of detergent material, such as saponified fatty acids or sulphonated organic compounds.

One of the objects of the present invention is to provide an improved method for increasing the apparent density of particulated detergents and the like.

Another object of the invention is to provide methods whereby the dusting of particulated detergents may be decreased.

A further object of the invention is to produce novel particulated detergents characterized by the high density of the product and the uniformity thereof.

These and other features of the invention will appear from the following description thereof, in which reference is made to typical examples for the purpose of indicating the nature of invention but without intending to limit the scope of the invention thereby.

In producing particulated detergents having high apparent density, in accordance with the present invention, the materials treated may be varied considerably. Thus, any of various synthetic, non-soap, organic detergents may be treated. Typical of such materials are water-soluble

salts of organic substituted, polybasic, oxygen-containing inorganic acids, including sulphates and sulphonates, borates and sulpho-borates, ortho-tetra-, pyro-, and polymetaphosphates, phosphites, etc. Numerous products of this character are described in U. S. Letters Patent No. 2,149,661; 2,149,662; 2,179,174; and Reissue Patent No. 20,636. In addition to such synthetic detergents, the invention is also applicable to treatment of spray dried soaps of various compositions, such as the sodium soaps of fatty acids. Such soaps, as well as the synthetic detergents, may initially contain fillers, builders, emulsifiers, wetting agents, buffers, plasticizers, or other modifying agents, if desired.

In treating any of these materials, the spray dried and porous particles may be treated directly at the time they are produced, or they may be treated subsequently to their production by passing the material into an agitating device such as a rotating drum, tumbler, vibrating conveyor belt, or other apparatus, wherein the material may be compacted or subjected to limited impacts without losing its particulated form. Within such apparatus, the material is subjected to the action of steam while continuing the rotation or agitation of the material. The steam apparently penetrates and softens or at least partially plasticizes the material, so that it is compacted and condensed during the agitation thereof, and the pores and interstices within the material are largely reduced or eliminated. The density of the product is thereby materially increased, so that a greater weight of the material may be contained within a unit of space, as a box, barrel or other receptacle. Spray dried synthetic detergents having an apparent density of 0.40 or more, and spray dried soaps having an apparent density of 0.50 or more, can be produced in this way.

Furthermore, during the agitation of the material, while in a softened or at least partially plasticized condition, the finer particles apparently become agglomerated with each other, or with the larger particles of the material, so as to form a product of more uniform size which contains very little fine material of the type which normally results in dusting of the product. Moreover, the tumbling or agitation of the material apparently wears down, or flattens out, or otherwise removes any weakened or projecting portions which might be broken from the larger beads or particles of the product when the materials handled or shaken about during the filling of containers and on shipment thereof. The material is, therefore, less friable and may be handled more readily without producing an objectionable amount of fines. At the same time, the tendency of the material to cake on standing is apparently reduced. It is also found that soaps treated in accordance with the present invention exhibit less tendency to form lumps when mixed with hot water and, as a result, the soaps dissolve more readily.

The amount of steam used and the length of time of treatment of the material will vary with the composition of the material and with the degree of apparent density desired in the finished product. In general, the application of steam to the material is continued until the moisture content has increased by about 3% to 20% based on the dry weight of the product treated. The material is then dried to reduce the total moisture content to that of the original material, or to any other desired point, preferably while continuing agitation of the material. In the case of synthetic detergents, the addition of steam preferably is continued until the product has increased in weight by about 3% to 7% before drying, whereas, when treating a spray dried soap, the amount of steam added may be sufficient to increase the weight of the product by from 10% to 50% before drying.

The temperature at which the atmosphere within the chamber is maintained during the tumbling or agitation of the material also may be varied, but generally it is in the neighborhood of from about 120° F. to 220° F., whereas the temperature of the particles may reach 135° to 155° F. or more after entering the chamber at an initial temperature of about 70° F. Steam has been used with great effectiveness, but moist steam, or a mixture of steam with the vapors or atomized particles of plasticizers such as glycerine, glycol or mineral oil, water-soluble sulphonated oils, or other modifying agents may be used, and the particles may be treated successively or simultaneously with steam and water or a coating, plasticizing or modifying agent.

In a typical operation, the material is passed through a rotating drum which preferably is inclined and into which steam is introduced, although any other apparatus or device which produces the same or similar agitation or movement of the particles may be employed. The treatment with steam is continued until the desired moistening of the product takes place, and the steaming then is discontinued or the product passed on beyond the steaming zone. It is then dried by passing warm, relatively dry air or inert gas over the material, preferably while continuing the tumbling operation. In the alternative, if desired, the material may be given an initial treatment with steam after which it is subjected to further and supplementary treatment with a spray of water or a solution of a coating composition plasticizer, modifying agent, or the like. The steam and liquid may be applied successively to the material, so that the treatment thereof with steam precedes a liquid treatment, or the application of steam may be simultaneous with the application of liquid, as desired. In any event, the length of time of treatment is relatively short, and the material can be dried more rapidly to reduce the moisture content of the finished product than when moistened with a spray of liquid alone.

In order to indicate typical procedure in accordance with the present invention, the following examples are cited by way of illustration, and not by way of limitation:

Example I

Particles of a spray dried synthetic detergent, 35% of which is the sodium salt of sulphuric acid ester of coconut oil fatty acid monoglycerides, and 65% of which is sodium sulphate, were selected for treatment. This product initially had an apparent density of about 0.25 gram per cubic centimeter, and contained from 1% to 2% of moisture. Approximately 55% of the particles of this product would pass an 80-mesh screen, and a portion of these fine particles was actually dust. Fifty pounds of this material having a temperature of about 70° F. were placed in a 55-gallon drum, rotating at about 11 R. P. M. and having an atmosphere maintained at a temperature of about 150° F. Dry steam was introduced into the drum for a period of 7 minutes, at the end of which time it was found that the mate-

rial had increased in weight by about 6%. Thereafter, while continuing to agitate the material, it was dried by warm air for a period of 15 minutes and allowed to cool. The resulting material had an apparent density of 0.40 gram per cubic centimeter, and a moisture content of 1.5%. The size of the particles was more uniform and generally larger than those of the product initially treated, and more uniform than those of a similar product which has been treated with a liquid spray in the absence of steam. The product contained relatively little fines and dusting material as compared with the material initially treated, as will be evident from the following table giving a more complete comparison of the size of the particles as determined by a screen test.

Screen Mesh	Untreated Product	Water Sprayed Product	Steam Treated Product
On 12	0.0	2.0	0.0
On 20	0.8	5.8	0.0
On 40	18.1	43.5	29.4
On 60	17.0	29.5	54.6
On 80	8.3	3.7	9.0
On 100	11.0	11.5	3.5
Pass 100	44.8	4.0	3.5

The material, when tested for caking, was subjected to a temperature of 90° F. and 90% relative humidity for a period of 72 hours, and then compared with a sample of the original product which had been similarly treated. The denser treated product was markedly less lumpy, and the lumps formed were so readily broken between the fingers that they could not be handled, whereas the lumps from the untreated product could be picked up and would not crumble without pressure. There was no apparent tackiness of the denser product, and it was found to flow and pour as readily as the untreated material.

Example II

Thirty-five pounds of a spray dried sodium soap having an apparent density of about 0.40 gram per cubic centimeter and containing approximately 10% of moisture and having an initial temperature of 70° F. were tumbled in a 55-gallon tumbler for 20 minutes at a temperature of 150° F., while maintaining an atmosphere of dry steam within the drum. Dry warm air was then passed through the drum, while continuing rotation thereof, for a period of 30 minutes and until the moisture content of the treated product was reduced to 12%. The apparent density of the material was thereby increased to 0.50, whereas the amount of fines or product capable of passing an 80-mesh screen was reduced as indicated by the following comparative table:

Screen Mesh	Untreated Product	Water Sprayed Product	Steam Treated Product
On 12	0.0	2.5	0.0
On 20	0.3	4.0	0.1
On 40	7.3	16.0	11.0
On 60	26.4	32.5	41.0
On 80	17.4	18.3	24.9
On 100	31.2	13.7	11.0
Pass 100	17.4	13.0	12.0

When subjected to a humid atmosphere at elevated temperatures, in a test for caking characteristics as described in Example I, the weight of the lumps of the treated soap particles was less than one third of the weight of the lumps produced in untreated soap subjected to the same conditions. Moreover, the product showed no evidence of tackiness, and the particles were generally more uniform in size and of better appearance. It was also found that lumping of the treated material in hot water was very substantially reduced. For purposes of comparing the lumping characteristics of the material, 15 grams of the dried product were added to 1 liter of water at 50° C. and allowed to stand for 1 minute. The mixture was then stirred at 1600 R. P. M. for 10 seconds, and decanted through a 10-mesh screen. The material retained by the screen was then weighed while wet, and it was found that 15 grams of the untreated material was retained by the screen, whereas only 2 grams of the steam treated material was retained by the screen.

This product is of particular interest because it possesses the desired high density and low moisture content without the usual high "builder" content of 25% or more which characterizes spray cooled soaps of similar apparent density. The product described above contained less than 10% of builder (soda ash in this case) and nearly 80% of fatty acids and unsaponifiable material, and therefore has a high soap content combined with high gravity (apparent density). Other soaps containing less than 25% builder and less than 25% moisture can be treated to produce a product having an apparent density of 0.50 or more.

Example III

A spray dried synthetic detergent, such as that treated in Example I, was introduced into a rotary drum while at a temperature of about 70° F., and steam was introduced into the drum so as to maintain an atmosphere therein at a temperature of about 150° F. The material was thus treated with steam while the drum was rotating until the moisture content of the product had been increased approximately 2% on the basis of the initial product. Thereafter, the treatment with steam was continued while introducing a finely atomized spray of water, and tumbling of the material was further continued until it had increased in moisture content about 6% further, making a total increase in moisture of about 8%. The application of steam and the liquid spray were then terminated and the material dried by passing warm air over the material while continuing to rotate the drum until the total moisture content in the product was reduced to 1½%.

The product thus obtained had a density, uniformity, and physical properties substantially the same as that obtained when treating the material with steam alone, as in Example I.

In each of the foregoing examples, the material may be treated with a solution of a coating composition or modifying agent for the purpose of increasing its solubility, altering its appearance or composition, adding a pigment or plasticizer, or for influencing the pH value or other properties of the treated material.

It will thus be seen that the present invention renders it possible to produce particulated detergents which have a much higher density than those heretofore produced by spray drying operations, and further serves to produce a product which is more uniform in size, has less tendency to cake or to form dust, and is more readily dissolved or dispersed in water. For example, spray dried soap having a relatively low moisture content, say less than 25%, may be treated in accordance with the present invention to obtain a product having an apparent density

of .50 gram per cubic centimeter or more, whereas particulated synthetic detergents, such as the salts of sulphonated higher fatty acid esters of glycols, may be produced which have a specific gravity up to 0.40 gram per cubic centimeter or more. Products of this character are novel and markedly advantageous over those of the prior art in that a greater weight of detergent may be stored and shipped in a given space, whereas the dusting of the material is reduced and the rapidity of solution thereof is increased.

While typical methods and products embodying the present invention have been described above, it should be understood that such methods and products have been cited for purposes of illustration only, and are not intended to limit the invention.

I claim:

1. A method of treating spray dried organic detergents which comprises the steps of tumbling particles of the detergent in an atmosphere of saturated steam until the total moisture content thereof has been increased by about 3% to 20% based upon the weight of the detergent, and thereafter drying the resulting detergent particles.

2. A method of treating spray dried organic detergents which comprises the steps of tumbling particles of the detergent in an atmosphere of saturated steam until the total moisture content thereof has been increased by about 3% to 20% based upon the weight of the detergent, and thereafter drying the resulting detergent particles while continuing agitation thereof.

3. A method of treating spray dried organic detergents which comprises the steps of tumbling particles of the detergent in an atmosphere maintained at a temperature between about 150° F. and 220° F. and in the presence of saturated steam until the total moisture content thereof has been increased by about 3% to 20% based upon the weight of the detergent, and thereafter drying the resulting detergent particles.

4. A method of treating spray dried organic detergents which comprises the steps of tumbling the particles of detergent in an atmosphere of saturated steam until the moisture content thereof has been increased by between 3% and 20% based on the weight of the detergent, while heating from a temperature of about 70° F. to a temperature of about 135° F. to about 155° F., and thereafter drying the resulting detergent particles.

5. A method of treating spray dried organic detergents which comprises the steps of tumbling the particles of detergent in an atmosphere of saturated steam until the moisture content thereof has been increased by between 3% and 20% based on the weight of the detergent, while heating to a temperature between about 70° F. and 150° F., and thereafter drying the resulting detergent particles.

6. A method of treating spray dried organic detergents which comprises the steps of tumbling the particles of detergent in an atmosphere maintained at a temperature between about 150° F. and 220° F. and in the presence of saturated steam until the moisture content thereof has been increased by between 3% and 20% based on the weight of the detergent, while heating to a temperature between about 70° F. and 150° F., and thereafter drying the resulting detergent particles while continuing agitation thereof.

7. A method of treating spray dried synthetic organic detergents which comprises the steps of tumbling particles of the detergent in an atmosphere of saturated steam until the moisture content thereof has reached about 3% to 7%, and thereafter drying the resulting detergent particles while continuing agitation thereof.

8. A method of treating spray dried organic detergents which comprises increasing the moisture content of spray-dried particles thereof by condensing steam thereon and by spraying water thereon until the particles are sufficiently plastic to be compacted by tumbling, and tumbling said particles until the apparent specific gravity has been increased to a desired value.

9. A method of treating spray dried organic detergents which comprises increasing the moisture content of spray-dried particles thereof by condensing steam thereon and by spraying water thereon until the particles are sufficiently plastic to be compacted by tumbling, and tumbling said particles until the apparent specific gravity has been increased to a desired value, and thereafter drying the particles.

10. A method which comprises the step of tumbling spray-dried particles of organic detergent while sufficiently plastic from exposure to saturated steam as to resist breaking, the tumbling being continued until the apparent specific gravity has been substantially increased.

11. A method of treating spray-dried organic detergent particles which comprises the step of exposing the particles to an atmosphere of saturated steam to heat the particles above about 70° F. but below about 220° F., subjecting said particles to impact while in a plastic condition resulting from said exposure to steam until the apparent specific gravity has been substantially increased and thereafter drying the particles.

12. A method of treating spray-dried organic detergent particles which comprises the step of exposing the particles to an atmosphere of saturated steam to heat the particles above about 70° F. but below about 220° F., tumbling said particles while in a plastic condition resulting from said exposure to steam until the apparent specific gravity has been substantially increased and thereafter drying the particles while continuing the tumbling thereof.

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