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DETERGENT FLUIDIZED DRYING PROCESS

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ABSTRACT OF THE DISCLOSURE

A process for drying wet particulate detergent material by feeding the material to a generally horizontal moving bed e.g., rotating drum, while passing heated gas upwardly through the bed in such a manner that a part of the bed is in a spout fluidized state. The process affords greater control over bulk density and particle size than the prior art methods.

This invention relates to a process for drying detergent materials containing an alkali metal polyphosphate. More particularly, it relates to a unique method for drying detergent materials wherein the bulk density and particle size of the dried material are easily controlled.

Spray drying, the most commonly used method for drying detergents, is limited to use of a slurry type feed which can be atomized into small droplets. Spray drying yields a product which is suitable for most detergent applications; however, it is a relatively expensive process and is not suitable for drying the porous relatively light density and fragile detergent products such as those described in U.S. Patent 3,177,147 and Belgium Patent 507,021. One of the advantages of the products described in the foregoing patents is their porous structure and their relatively light density. It has been found, however, that conventional drying equipment is unsuitable in most instances because the bulk density and particle size undergoes a substantial change during the drying process. For example, conventional rotary driers, wherein a heated gas stream flows parallel to a bed of material inside a rotary drum, are unsuitable because a large amount of lumps are formed and the bulk density is increased by a sizeable amount. The rolling action of the particle in the bed and compaction pressure of the bed cause layers of wet detergent material to form on the particles and results in lumps and a sizeable density increases. Vertical fluidized bed driers, wherein a flow of heated gas passes upwardly from the bottom of a bed through the bed, are unworkable because variations in the particle size and densities of the wet feed detergent material result in plug-ups and/or the generation of large quantities of unsuitable fine particles from attrition. The gas flow rate in a vertical fluidized bed drier is limited by keeping a bed of material in a fluidized state. If the sizes and/or densities of the particles vary beyond the desired range, the larger or heavier particles become concentrated at the bottom of the bed and results in plug-up because the gas flow is insufficient to keep the bed in a fluidized state. If the gas flow is increased so that the large particles can be fluidized, an undesired amount of the smaller particles will be carried out in the exiting gas stream. Additionally, at the high gas velocities the fines generation is increased resulting from the attrition between the particles.

It is believed, therefore, that a convenient process for drying a light density, relatively fragile detergent material of varying particle sizes and densities wherein the bulk density and particle size of the dried material are easily controlled would be an advancement in the art.

In accordance with this invention, it has been found that the bulk density and particle size of relatively light density and fragile detergent materials containing an alkali metal polyphosphate can be controlled during drying by a process comprising preparing a wet, particulate detergent feed material containing from 10 to 35% water by weight; feeding the wet detergent material to a substantially horizontal moving bed of detergent material; passing a gas stream upwardly through a portion of said bed and substantially normal to the movement of the bed thereby removing a portion of the water from the detergent material; discharging from the bed a detergent product containing from about 2 to about 15% water by weight and maintaining the apparent velocity of the gas entering the bed, feed rate, and product discharge rate so that a portion of the bed is in a "spout-fluidized" state.

Particle size change and density change is controlled by the gas stream passing upwardly through the bed which causes at least some of the particles to be entrained in the gas stream for a short distance above the bed. This effect, referred to herein as "spout fluidization" is achieved by using gas flow rates which create certain apparent velocities at the point of entering the bed which are related to the particle size and densities of the material to be dried. Spout fluidization reduces the compaction pressure and the rolling action thereby obviating the density increase and lump formation obtained in conventional rotary driers. A simple means for determining if spout fluidization is occurring is to observe the bed. The bed appears to be in a "spouting" state; that is, the gas stream flowing upwardly through the bed creates the illusion that the bed is boiling and is being showered with particles because some of the particles are entrained in the gas stream for a short distance after the gas leaves the bed but then drop back to the bed because the gas stream velocity decreases after passing through the bed. The foregoing simple observations can be used on existing installation satisfactorily; however, this means is not practical for other purposes, such as the design of new equipment. It has been found, however, that conditions of spout fluidization in the bed are related to the apparent velocity of the gas stream entering the bed and to the particle size and density of the wet detergent feed material. Therefore, for a particular proposed drying system an apparent velocity of the gas stream as it enters the bed can be calculated. The apparent velocity of the gas stream entering the bed is the total gas flow per unit of time divided by the area of the gas distribution device through which the gas flows just before the gas enters the bed of detergent material.

The apparent velocity which will render a portion of the bed to a spout fluidized state is that velocity which will theoretically lift some of the particles of the wet materials, that is, the velocity exerts a lifting force mathematically equal to the weight of a given particle. Thus, the minimum gas velocity which will lift a particle can be determined from a force balance, i.e., weight of particle=lifting force from the gas stream or

$$\left(\frac{\pi D^3}{6}\right)(\rho s)=\left(\frac{\pi D^2}{4}\right)(\rho a)\frac{V^2}{2g}$$

$$V=\frac{4}{3}\,\frac{\rho s D g^{0.5}}{\rho a} \qquad \text{Formula I}$$

wherein, in consistent units,

V=superficial gas velocity, ft./sec.
ρs, ρa=density of particles and gas respectively, lb./ft.^{3}
D=diameter of particle, ft.
g=gravitational constant, 32.2 ft./sec.^{2}

For a particle diameter equal to the opening of a U.S. Standard 12 mesh screen, an apparent solid particle density of about 30 lb./ft.^{3}, and a gas density of about 0.048 lb./ft.^{3}, the gas velocity required to lift a particle will be:

$$V=\frac{4}{3}\times\frac{30}{0.048}\times\frac{0.066}{12}\times 32.2^{0.5}=12.2 \text{ ft./sec.}$$

Although some of the beneficial results from this invention can be achieved by using apparent velocities sufficient to lift only about the smallest 1% of the particles in a wet detergent feed, it is generally preferred to use an apparent velocity which would have a lifting force theoretically sufficient to lift from about 30% to about 90% of the particles in the wet detergent feed material. It is further preferred when a minimum density increase is desired to use velocities sufficient to lift from about 40% to about 80% of particles in the wet detergent feed. Similarly, some of the beneficial results can be achieved by employing apparent velocity which will lift all but about the largest 1% of the particles in the segment of the wet feed material which is being subjected to the upward gas flow. In most instances, however, it is preferred to use the before-mentioned ranges of apparent velocities.

"Upwardly," as used herein, means that as the gas stream passes through the bed, it exerts a lifting force on the particles in the bed. "Upwardly," as used herein, is not to be equated with "vertically" because in some instances such as when a rotary type of drier is used in the practice of this invention, the surface of the bed is canted from the horizontal and the air distribution devices such as the louvers, fins, and the like can direct the flow of air as it enters the bed in a substantially horizontal direction. Of course, when the surface of the bed is in the horizontal plane the gas can flow in a vertical direction. The value of the apparent velocities that will be used will depend upon the particle size of the wet detergent feed. For example, when drying a typical detergent material having an average particle size ranging from about −8 mesh to about −60 mesh and having 90% smaller than about +2 mesh and 90% larger than about −100 mesh, a 290° F. air stream having an apparent velocity of 10–25 ft./sec. reduces the moisture content of a wet detergent material from about 22% water to about 10% water and with an increase in bulk density of less than 0.05 g./cc. and with a change in particle size of less than about 5%.

By regulation of the apparent velocities of the gas stream entering within the ranges herein specified, the bulk density of the material leaving can be readily controlled. The lower velocities, that is, apparent velocities of the gas stream entering which will exert a force sufficient to lift less than 50% of the particles in the feed will yield a greater density increase than the higher velocities, that is, those which exert a force sufficient to lift greater than 50% of the particles in the feed. This flexibility cannot be obtained in a vertical fluidized bed drier in which the gas velocity must be high enough to keep all particles in satisfactory motion.

To achieve the benefits of this invention, it is only necessary that a small portion, that is, as little as 5% or even less such as 2% of the bed, be in a spout fluidized state. It is preferred, however, in most instances that from about 20% to about 100% of the bed be in a spout fluidized state. It is preferred that the hot gas stream flow concurrent with the flow of the feed material so that the material with the highest water content is contacted with the highest temperature gas stream. When using concurrent material and gas flow and less than 60% of the bed in the spout fluidized state, it is preferred that a major proportion of the spout fluidization occurs in the 50% of the bed closest to the point where the wet detergent feed material enters.

As mentioned previously, the wet detergent material is fed to a horizontal moving bed. A simple means for providing a horizontal movement is to use a horizontal but slightly canted rotary drum where the gas stream enters underneath the bed through louvers, fins, or other distribution devices and then passes through the bed in a direction substantially normal to the movement of the bed. The moving bed eliminates the accumulation of large particles which cannot be fluidized by the air-jet stream.

The depth of the bed in a rotary drier is relatively unimportant; however, if it is desired to minimize the density increase, a relative shallow bed is preferred. The density increase, even with deeper beds, is considerably less than that experienced with other driers. For example, with a wet detergent product having a density of about 0.3 to 0.35 g./cc., essentially no increase is observed at a 0.1 inch bed depth, from 0.02 to 0.03 g./cc. increase at 2 to 3 inches, and both a 4- and 6-inch bed depth gives only an increase of from 0.03 to 0.05 g./cc. In a conventional rotary drier using similar detergent material an increase of from about 0.20 to about 0.35 g./cc. is obtained. As used herein, "bed depth" refers to the maximum depth of material at the feed end of the drier. It has also been found that a preferred gas distribution is obtained with shallow beds when a rotary type drier is used, that is, a larger proportion of the gas passed through the bed at the feed end, thus the wet detergent particles are quickly converted from a sticky material tending to agglomerate to a relatively free-flowing material. Therefore when a density increase of less than about 0.03 g./cc. is desired, the preferred bed depth is from about one inch to about three inches, while if a density increase of 0.05 g./cc. can be tolerated, a bed depth of from 1 to 6 inches can be used.

The pressure drop across the drying bed will be dependent upon the bed depth, gas flow and design of the system. In most instances, the pressure drop is relatively low; that is, from less than about 1 to about 5 inches of water. However, satisfactory results can be obtained with pressure drops as high as 10 to 15 inches of water.

The sojourn time of the material will also vary depending upon the particular design and operating conditions chosen. In most instances, however, particularly when the density, increase is held to a minimum by utilizing a shallow bed depth, the sojourn time is relatively low. For example, on a 26″ diameter drier with less than a 2″ bed depth, utilizing 660 c.f.m. of air (corrected to 70° F.) at 410° F. with a feed containing 18.5% water, and a bulk density of from 0.3 to 0.35, a desirable product containing about 10–12% water with a density increase of about 0.03 g./cc. was produced in a rotary drier under spout fluidization conditions with a sojourn time of about 5 minutes.

One of the preferred embodiments of this invention is to dry light density detergent materials such as those produced by the processes disclosed in Belgian Patent 507,021 or U.S. Patent 3,177,147. It has been found that the light density detergent materials (those having a bulk density below about 0.7 g./cc. and especially those from about 0.20 g./cc. to about .45 g./cc.) are extremely difficult to dry without a sizeable density increase due to the agglomeration or compaction and/or by physical degradation of the particles. By practicing this invention, a light density wet detergent material, that is, one below 0.7 g./cc. and particularly those having a bulk density of from 0.20 g./cc. to about 0.45 g./cc. detergent material can be dried without an appreciable density increase and produce a dried detergent product substantially free of −100 mesh and +2 mesh particles thus offering a distinct advancement over any heretofore known methods for drying these products. This advantage is clearly illustrated in Example 2.

As previously mentioned, wet detergent material containing from about 10 to about 35% water can be dried by the process of this invention. Generally in most processes, the detergent material will have contained from about 25 to 32% water. The water content as used herein refers to total water content, that is, it includes both water of hydration or water of crystallization and free water in the detergent material. In most instances the amount of free water in the wet detergent material will range from 2% to about 22% with a corresponding range of total water content from about 10 to about 35%.

In general, this invention can be used to reduce the water content of most detergent materials; however, since the air velocity required for lifting large particles will reach a practical limit, it is desired to have a particle size sufficiently small so that at least 50% will pass through a U.S. Standard 2 mesh screen. It is even more preferable to use a wet material which has at least 90% of its particles smaller than theopening in a U.S. Standard 2 mesh screen. It is generally desired not to have excessive amounts of extremely small particles in most commercial detergents; that is, it is preferred that the amount of dried material which would pass through a U.S. Standard 100 mesh screen be less than about 20% by weight. It is, however, more particularly preferred that the wet material utilized in this invention contain less than 15% by weight of particles which will pass through a U.S. Standard 100 mesh screen to thereby produce a more desirable product and to minimize dust losses. In most applications, it is desirable to have the average particle size in the range of from about −8 mesh to about +60 mesh in the wet detergent feed material. It is a unique advantage of this process that using a wet detergent feed larger than preferred size, the drier will not plug-up as opposed to the vertical fluidized bed driers.

The process of this invention can be used to dry any detergent material containing at least 0.5% by weight of alkali metal polyphosphate based upon the wet detergent feed material on an anhydrous basis and containing from about 10% to about 35% water and which has a bulk density on a dry basis less than 0.7 gram per cc. To obtain these compositions, it is generally necessary in the production to use a similar process to those described in the before-mentioned patents. The process of this invention can be used to dry detergent materials which are complete formulations as well as those which are subsequently blended with other ingredients to form formulations that are desired for particular uses. For example, a detergent base composition can be prepared as in Example 5 and subsequently blended with surfactants and other raw materials such as chlorinated cyanuric acid or cyanurates to form a desired formulation. It is generally preferred that the alkali metal polyphosphate be sodium tripolyphosphate because of its relative inexpensiveness. It is also preferred that the sodium tripolyphosphate be in the hexahydrate form such as that produced from the reaction of sodium trimetaphosphate and a strong base such as sodium hydroxide. In most instances, a surface active agent will normally be employed in the detergent material. Although as little as 0.5% by weight based upon the wet detergent material on an anhydrous basis of the alkali metal polyphosphate can be present, greater amounts will normally be present such as from about 10 to about 80% by weight based upon the amount of polyphosphates and surface active agent present on an anhydrous basis. In most instances when a relative complete formulation is dried, the detergent material will be a mixture containing a surface active agent of the classes hereinafter described and an alkali metal polyphosphate. The weight ratio of the surface active agent to polyphosphate in the formulation will generally range from about 1:5 to about 5:1.

The surface active agents which are generally employed are various soaps such as those produced from the saponification of a fatty acid such as palmitic, oleic, and the like and the synthetic organic surfactants including the anionic, nonionic, and amphoteric types and mixtures thereof.

Anionic synthetic surface active agents are generally described as those compounds which contain hydrophilic and lyophilic groups in their molecular structure and ionize in an aqueous medium to give anions containing both the lyophilic group and hydrophilic group. The alkyl aryl sulfonates, such as sodium dodecylbenzene sulfonate; the alkane sulfates, such as sodium dodecyl sulfate; and the sulfated oxyethylated phenols, such as sodium tetradecyl phenoxy triethyleneoxy sulfate, are illustrative of the well-known class of anionic type of surface active compounds.

Nonionic surface active compounds can be broadly described as compounds which do not ionize but acquire hydrophilic characteristics from an oxygenated side chain such as polyoxyethylene and the lyophilic part of the molecule may come from fatty acids, phenol, alcohols, amides, or amines. The compounds are usually made by reacting an alkylene oxide such as ethylene oxide, butylene oxide, propylene oxide and the like with fatty acids, the straight or branched chain alcohols, phenols, thiophenols, amides, and amines to form polyoxyalkylene glycol ethers and esters, polyoxyalkylene alkyl phenol and polyoxyalkylene thiophenols, and polyoxyalkylene amides and the like. It is generally preferred to react from about 3 to about 30 moles of alkylene oxide per mole of the fatty acids, alcohols, phenols, thiophenols, amides, or amines. Illustrative of the surface active agents include the product obtained from condensing ethylene oxide with the following: propylene glycol, ethylene diamine, diethylene glycol, dodecyl phenol, nonyl phenol, and the like.

Amphoteric surface active compounds can be broadly described as compounds which have both an anionic and cationic group in their structure. Illustrative of the amphoteric surface active agents are the amido alkane sulfonates, such as sodium C-tridecyl, N-methyl, amido ethyl sulfonate.

Other individual compounds which are illustrative of the foregoing classes of surface active agents are well known in the art and can be found in standard detergent reference materials such as Surface Active Agents, Swartz and Perry, Interscience Publishers, Inc., New York, N.Y. (1949).

The detergent additives which can be employed in detergent composition formulations that can be dried by the process of this invention include various organic and inorganic salts and mixtures thereof. Generally, these additives constitute from about 10 to about 85% by weight of the detergent composition and add to the detergency efficiency of the surface active agent.

The organic compounds which are used included aminopolycarboxylic acids, their water soluble salts, such as nitrilo triacetic acid and its alkali metal salts; the amino tri(lower alkylidenephosphonic acids) and their water soluble salts such as amino tri(methylenephosphonic acid) and its alkali metal salts and the alkylene diphosphonic acids and their water soluble salts such as methylene diphosphonic acid and its alkali metal salts and mixtures of the foregoing compounds.

The inorganic salts which are normally used in detergent compositions include the alkali metal polyphosphates and in particular the sodium and potassium tripolyphosphates and pyrophosphates. The detergent materials will contain an alkali metal polyphosphate and particularly it is preferred that some sodium tripolyphosphate be present, generally in amounts of from at least about 5% to about 80% by weight with amounts from 20% to 60% by weight being especially preferred.

Additionally in many of the detergent compositions other additives are present such as antiredeposition agents, brightening agents, corrosion inhibitors, perfumes, inert fillers, dyes, bluing agents, and the like. Typical examples of such additives are sodium carboxymethyl cellulose, polyvinyl alcohol, sodium sulfate, sodium silicate, methyl cellulose and sodium carbonates.

The gas streams that can be used in the practice of this invention are, in general, any gas stream which will remove some of the free water from the detergent material and which will not adversely affect the material either by excessively high temperature or by reaction with any of the components contained therein. Normally, air or flue gas will be used; however, other inert gases which will absorb water from the wet detergent material can be used, such as CO_2, nitrogen, and the like. Selection of the particular gas stream used will be dependent upon the material that is to be dried. In most instances, heated air will be satisfactory and is preferred. It is to be noted that although any of the foregoing gas streams which have less than 100% relative humidity can be used, it is preferred to use gases having lower relative humidities, that is, below 50% or even lower, that is below 20%. In most instances if a heated gas stream is used, that is, one from about 220° F. to about 550° F., humidity is not a problem since normally these gases will not be saturated if the conventional methods of heating the gases are employed. In this case, the humidity of the exhaust gas stream should be less than 95% saturated so that condensation will not take place in the exhaust ducts. If, however, it is desired to use a gas stream having a temperature below about 220° F., it is preferred to use gas streams containing the before-mentioned lower relative humidities.

Any gas stream temperature can be used which will not result in the degradation of the detergent composition. In most instances and with most detergent compositions temperatures of up to about 620° F. can be employed without degradation of the detergent components unless some particularly heat senstive component is incorporated therein. It is preferred to use entering air temperatures of from about 250° F. to about 600° F. in most cases with from about 300° F. to about 560° F. being, particularly preferred for most light density detergent materials.

The outlet gas temperature will vary depending upon several variables, such as water to be removed from the feed, flow of air, particle size, and bed depth. Under the preferred conditions, about 100° F. to 300° F. drop in temperature of the air can usually be obtained. Under the preferred conditions, it is also possible to obtain relatively high thermal efficiencies, that is, about 50% or even higher such as 60% to 70% with proper insulation and minimum of heat loss.

As mentioned previously the dust loading in the exiting gas stream is lower than many of the conventional drying processes particularly when the gas stream velocity is within the preferred range and will normally represent less than a 2% loss of product. If desired, conventional dust recovery systems such as scrubbers, cyclone separators, and the like can be used as auxiliaries.

The temperature of the detergent product will depend upon several variables, such as, temperature of the feed, temperature of the gas stream, sojourn time and water content of the dried material. In most instances, however, the temperature of the product discharged from the drier will be only slightly cooler than the outlet temperaure of the gas stream, that is, about 5 to 20 degrees F. below the temperature of the outlet gas stream. When sodium tripolyphosphate hexahydrate is present, this invention is particularly suitable since the product temperature can be controlled at temperatures below 160° F. to reduce dehydration of the hexahydrate. Other methods of drying tend to dehydrate the hexahydrate and cause degradation. Thus, it is possible by employing this invention to produce a detergent product which has a sodium tripolyphosphate assay about 10% higher than would be achieved in a convenional spray drying detergent process. As can be appreciated where the material being dried contains large amounts, that is, from 30 to 90% by weight of sodium tripolyphosphate hexahydrate, this lack of degradation is of extreme importance.

As previously mentioned, the particle size and bulk density of the detergent product can be controlled by varying the bed depth and/or the apparent velocity of the entering gas stream. In most instances, it is desired to operate with a minimum change in bulk density and particle size. Thus, under preferred operating conditions as specified herein a bulk density change of less than about 0.05 g./cc. can be achieved and under low bed depth conditions the bulk density change can be less than 0.03 g./cc. compared to the bulk density of the wet detergent material. Similarly, there is little change in the overall particle size since agglomeration and attrition is minimized; thus the overall change in particle size under the preferred operating conditions is less than 15% and normally will be from 5 to 10% compared to the particle size of the wet detergent material.

To specifically illustrate the invention, the following examples are presented. All parts, proportions and percentages are by weight unless otherwise indicated.

Example 1

About 300 parts of sodium dodecylbenzene sulfonate, about 2 parts sodium toluene sulfonate, about 100 parts of a 40% aqueous solution of sodium silicate, about 260 parts of sodium sulfate, about 110 parts of water, and about 360 parts of sodium trimetaphosphate are charged into a jacketed mixing vessel. The mixture is agitated and heated to about 70° C. by circulating steam in the jacket of the mixing vessel and is then pumped into an in-line mixer at the rate of about 20 parts per minute.

About 310 parts of a 50% sodium hydroxide solution and about 500 milliliters of air per minute are added to the in-line mixer. The mixture from the in-line mixer is discharged onto a reaction belt in the form of a slurry. The sodium trimetaphosphate and sodium hydroxide start to react in about one minute with the evolution of heat and steam. The temperature of the reaction mass reaches about 105° C. and the material forms a foamed solid. The conversion of the trimetaphosphate to tripolyphosphate by the sodium hydroxide is essentially complete in about 10 minutes. The foamed mass contains about 27% water and is easily disintegrated into a particulate mixture containing particles ranging from about 1 inch in diameter to less than the opening in a U.S. Standard 100 mesh screen. About 80% of the detergent material has a particle size smaller than the openings in a U.S. Standard 8 mesh screen and larger than the openings in a U.S. Standard 100 mesh screen and has an apparent bulk density of about 0.3 g./cc.

Example 2

Detergent materials prepared as in Example 1 are dried using three processes identified hereinafter as Process No. 1, Process No. 2, and Process No. 3.

In Process No. 1 the detergent material is dried using a vertical fluidized bed drier using an air temperature of about 150° C. In this vertical drier, the hot air passes through the bed at a velocity theoretically sufficient to keep the entire bed well agitated and in suspension. The product is suspended in the gas stream until dried, then separated from the exiting gas stream.

In process No. 2, the detergent material is dried using a conventional rotary drier with an inlet air temperature of about 180° C. In this drier a bed of material is placed in a rotary drum and the air flows parallel to the drying bed.

In Process No. 3, the detergent material is dried using the process of this invention using a rotary type drier with air having a temperature of about 360° F. passing upwardly through louvers under the bed to give direction to the air flow through the bed.

The following table lists the characteristics and the results from the utilization of the three processes.

TABLE I

Characteristics	Process No. 1	Process No. 2	Process No. 3
Product:			
Moisture content, percent	11–13	12–16	12–13.5
Apparent density, g./cc.	[1] 0.35–0.55	0.50–0.65	0.30–0.37
Particle size dist., percent:			
+8 mesh	10	65	10
−100 mesh	15	5	4
Build-up in the drier	[2]	[3]	[4]
Dust in Exhaust, percent of total products	5–15	3–10	3–5
Superficial air velocity, across or above the drying bed, ft./sec.	4–6	3–4	[5] 13–17
Inlet Air Temp., ° F.	[6] 300	360	360
Outlet Air Temp., ° F.	140	[7] 190	155
Product Temp., ° F.	122	154	150
Sojourn, Min.	5–15	10–20	5–10
Est. percent $Na_5P_3O_{10}.6H_2O$ degraded	1–5	3–8	1–

[1] Product density from the fluidized-bed drier usually shows an increase with time. This is due to attrition between particles in the fully agitated bed.

[2] Plug-up after 2–3 hours operation.

[3] Drier surface is coated. Large wet balls are formed in the drier.

[4] Clean metal surface. No sign of build-up.

[5] Velocity leaving louvers under the bed.

[6] Lower inlet gas temperature is used to increase total air rate for fluidization.

[7] Due to significant decrease in heat and mass transfer rates from the formation of dense balls, the drier product and exhaust gas temperatures increase.

As can be readily seen, the process of this invention (Process No. 3) is superior to either of the conventionally used processes in density control, operation, and particle size distribution, thus clearly illustrating the benefits from this invention.

Example 3

About 160 parts of water, 125 parts of an aqueous sodium silicate solution containing about 44% solids, about 2 parts sodium toluene sulfonate, 10 parts of commercial sodium carboxymethyl cellulose and 1 part of an optical brightener are thoroughly mixed in a ribbon blender. About 270 parts of tridecylbenzene sulfonate are added to the mixer along with about 58 parts of sodium sulfate. These materials are mixed in a uniform slurry which is heated to a temperature above 45° while mixing. About 34 parts of sodium tripolyphosphate are then added to the mixture and thoroughly blended within the space of about 1 minute, following which about 17 parts of 35% solution of hydrogen peroxide are added to the mixture and mixed for about 30 seconds. Immediately after the paste is dumped from the mixer into a vat having a volume approximately 3 times that of the material prior to expansion. The material in the vat is permitted to stand at 25° C. and during the 15 minutes the material swells to about 2 to 3 times its initial volume and the temperature rises to about 65° C. The wet detergent material is permitted to cool to about 35° C. and then exposed to an ambient temperature of about 25° C. for about 48 hours. The material is then removed and placed in a rotating cage mill and then screened through a sieve to reduce the particle size. Essentially all of the material passes through a U.S. Standard 4 mesh screen after screening. The screened particles contain from about 22 to about 23% moisture and are conveyed to a rotating drum type drier to form a bed. Air heated to about 320° F. is used to dry the material. Two methods of air passage are used. In the first method, which is conventional, the air passes concurrent with and over the solids. In the second method, according to the present invention, the air passes through distribution louvers underneath the bed for an air velocity at the openings about 20% greater than that required to lift substantially all of the particles thus creating a spouting and showering bed near the feed end. The product from the first method contains more fines with a density increase from about 0.3 to about 0.4 g./cc. Using the second method, the process of the present invention, the material discharging from the drier shows little change in the fines content and the apparent bulk densities of the entering material and the leaving material are essentially the same at about 0.3 gram per cc. The material entrained in the exiting air is measured at less than about 2% of the wet material entering the drier.

Example 4

Into a jacketed stainless steel mixing vessel which is fitted with the conventional type stirrer, a funnel shape extension above the vessel for reaction means to expand, and connectors to the jacket so that either hot or cold water or steam can be used in the jacket are charged 2,000 parts of water, 900 parts of sodium dodecylbenzene sulfonate, 600 parts of sodium lauryl sulfate, 40 parts of sodium toluene sulfate, and 1,000 parts of sodium sulfate and 2,840 parts of sodium trimetaphosphate, 1,100 parts of sodium silicate, and 55 parts of detergent grade of sodium carboxymethyl cellulose. The resulting slurry is stirred for about 20 minutes during which time the temperature of the slurry is raised to 80° C. by circulating steam through the mixer jacket. Into the hot precursor slurry which is being vigorously stirred is then quickly poured about 1,600 parts of 50% sodium hydroxide solution. After about 45 seconds of continuous agitation during which time the sodium hydroxide is blended well with the slurry, the temperature of the slurry begins to rise. The agitation is halted just before the temperature of the slurry is 100° C. When the slurry temperature reaches about 105° C. the slurry begins to expand in volume from escaping steam. The volume of the foamed mass is considerably greater than 3 times the volume of the reaction slurry. The foamed mass hardens and easily breaks into a particulated wet and fragile mass. The material is discharged into a conveyer belt after aging 5 to 15 minutes in the vessel and the extension piece. The material is cooled on the belt and introduced into a vibrating conveyer through which a hot air stream passes upwardly through the wet material. The material as charged to the conveyer belt has an apparent bulk density of about 0.37 gram per cc. and with a particle size of about 80% passing through a U.S. Standard 8 mesh screen. The apparent air velocity entering the bed of wet material is regulated to above 8 feet per second so that the particles are periodically suspended by the air jets. The material is dried to give a total moisture content of about 11%. The larger particles, that is, those having a particle size larger than a U.S. Standard 8 mesh screen, are easily granulated by passing through an 8 mesh screen. The combined product streams with a density of about 0.35 g./cc. are packaged for market. It is of interest to note when the apparent velocity of the entering air is below that required to lift any of the particles, the wet product balls up and yields a product having a density of greater than about 0.5 g./cc. which dissolves much more slowly.

Example 5

A precursor slurry is prepared in a standard agitated reaction vessel by adding 290 parts sodium sulfate, 2 parts of sodium lauryl sulfate, 300 parts of sodium trimetaphosphate and 250 parts of water and heating to about 70° C. After the temperature of the precursor slurry reaches about 70° C. about 148 parts of a 50% aqueous sodium hydroxide solution are added. An exothermic reaction occurs causing the temperature to rise to about 105° C. with an evolution of steam. After about 10 minutes the material forms a porous mass containing about 30% water which is granulated through a U.S. Standard 8 mesh screen. The bulk density of the product on a 70% moisture basis is about 0.43 g./cc.

The material is dried using two methods, Method A and Method B. In Method A the material is charged into a rotary drum to form a bed. An air jet having an air temperature of about 400° F. is used to provide an entrance velocity into the bed which is sufficient to lift about 60% of the particles as it passes upwardly through the bed but the exit velocity after leaving the bed is below that which entrains more than 2% by weight of the particles in the bed. The product discharged has a bulk density of about 0.45 g./cc. and a water content of about 13%.

In Method B the same temperature air stream is used, however, the air enters parallel to the bed and passes over it as in a conventional rotary drier. The inside of the rotary drum is coated with material and the material forms several large lumps. The bulk density of the material after drying to a water content of about 13% is about 0.65 g./cc.

About 3 parts of the product from Method A are blended with one part of potassium dichlorocyanurate to form an excellent dry bleach composition. Since essentially all of the water is bound as the hexahydrate of sodium tripolyphosphate, a good stability of the chlorinated cyanurate is achieved.

What is claimed is:

1. In the process for manufacturing a dried porous particulate detergent material containing an alkali metal polyphosphate and having a bulk density less than about 0.7 g./cc. wherein a wet particulate detergent material feed containing from about 10% to about 35% water by weight is prepared and is subsequently heat dried, the improvement comprising feeding said wet detergent material to a substantially horizontal bed, initially mechanically moving said bed of detergent material; passing a heated gas stream upwardly through a portion of said bed and in a direction substantially normal to the movement of said bed to thereby remove a portion of the water from said detergent material; discharging from said bed a dried detergent material having a total water content of from about 2% to about 15% by weight and maintaining the apparent velocity of the gas entering the bed, feed rate and discharge rate so that at least a portion of the bed is in a spout fluidized state.

2. A process according to claim 1 wherein at least 90% by weight of the particles in said material in said bed pass through a U.S. Standard 2 mesh screen and less than 20% by weight of said particles pass through a U.S. Standard 100 mesh screen and said gas stream is air having an entrance temperature of from about 250° F. to about 600° F. and wherein from about 20% to about 100% of said bed is in a spout-fluidized state.

3. A process according to claim 2 wherein said alkali metal polyphosphate is sodium tripolyphosphate hexahydrate and is present in amounts of at least about 0.5% by weight based upon the wet detergent material on an anhydrous basis.

4. A process according to claim 3 wherein said bulk density of said dried detergent material is from about 0.2 g./cc. to about 0.45 g./cc.

5. A process according to claim 4 wherein said apparent velocity of said air stream is sufficient to lift from about 30% to about 90% of the particles in said wet detergent feed material.

6. A process according to claim 5 wherein the bulk density of said wet detergent feed material changes less than about 0.05 g./cc. during said process.

7. A process according to claim 6 wherein said wet detergent material contains a surface active agent selected from the group consisting of anionic, nonionic and amphoteric synthetic detergents and mixtures thereof and the ratio of surface active agents to sodium tripolyphosphate is from about 1:5 to about 5:1.

8. A process according to claim 7 wherein said sodium tripolyphosphate is produced from the reaction of sodium trimetaphosphate and sodium hydroxide.

9. A process according to claim 8 wherein the depth of said bed is from about 1 to about 6 inches and wherein the average particle size of the wet detergent feed material is from about −8 mesh to about +60 mesh and wherein the pressure drop across said bed is from about 1 to about 5 inches of water.

10. A process according to claim 8 wherein said sodium tripolyphosphate is present in said detergent material in amounts of from about 10 to about 80% by weight based on the amount of polyphosphates and surface active agent present on an anhydrous basis.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,426,440 Dated February 11, 1969

Inventor(s) Chung Yu Shen & Norman Earl Stahlheber

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Colum 9, line 15, under Process No. 3, reading "1-" should read -- 1-5 --.

SIGNED AND
SEALED
AUG 11 1970

(SEAL)
Attest:

Edward M. Fletcher, Jr.
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Commissioner of Patents