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[54] **DRYING OF DETERGENTS**

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[51] **Int. Cl.**..**F26b 3/00**
[58] **Field of Search**.......................................34/9, 12

[56] **References Cited**

UNITED STATES PATENTS

500,535	6/1893	Des Cressonnicies	34/12
725,363	4/1903	Powter	34/12

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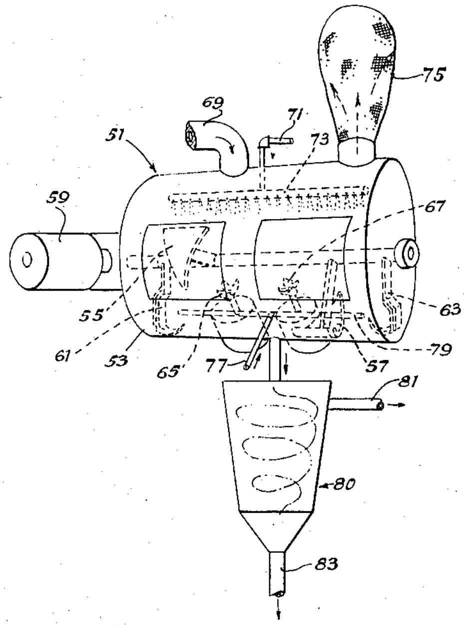
[57] **ABSTRACT**

Aqueous solutions of heat sensitive synthetic organic detergents, such as sodium higher alkyl benzene sulfonates, are dried by admixing such a solution with a very finely divided sorbent material, such as a water soluble builder or filler salt or an insoluble scouring material, thereby distributing the solution over a very greatly increased area, which speeds drying and prevents hot spots during drying, and drying the mixed detergent-sorbent-moisture particles.

10 Claims, 3 Drawing Figures



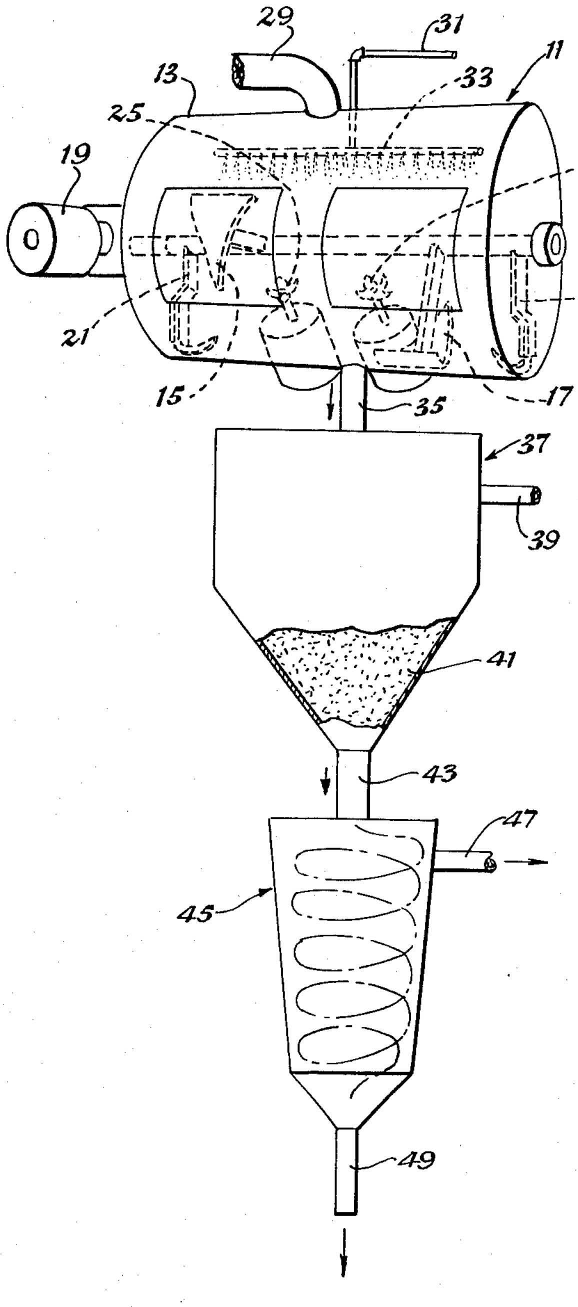


Fig.1.

Fig.2.

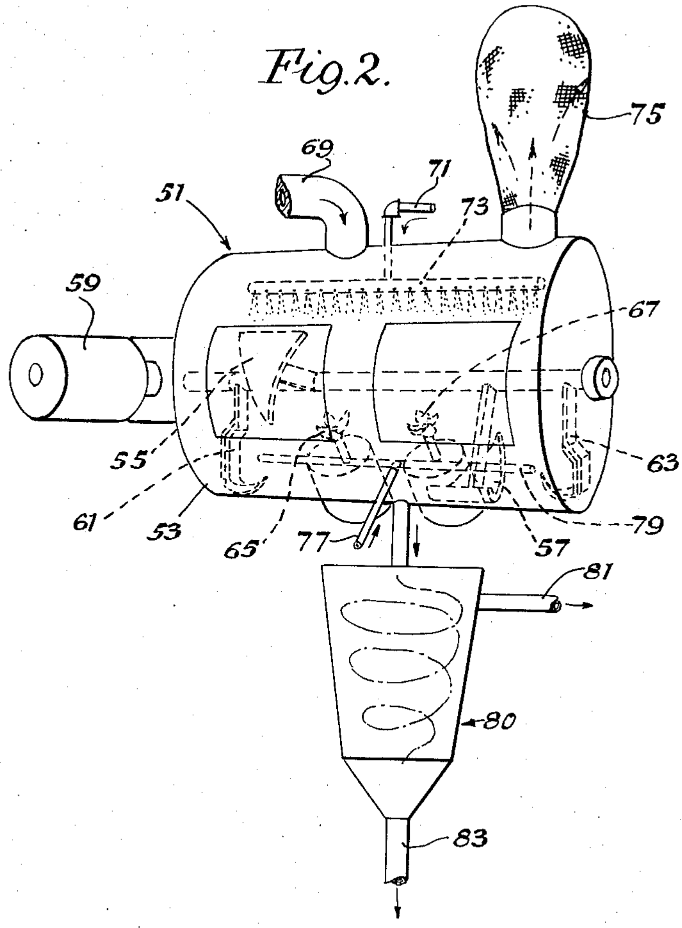

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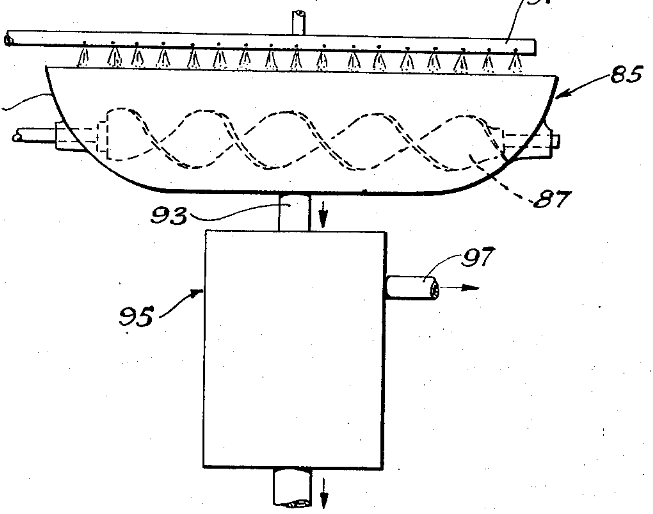
Fig.3.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DRYING OF DETERGENTS

This invention relates to a method for drying aqueous solutions of synthetic organic detergents, especially those which are heat sensitive. The drying is effected by first distributing the detergent solution over the surfaces of a particulate sorbent, which may also serve as a component of a detergent composition or a composition containing such detergent, and then removing the moisture from the particles by evaporation with a drying gas at a temperature at which the detergent is stable.

Many detergents are produced by chemical reactions that result in solutions which must be evaporated to obtain the detergent in solid form. After such a drying operation, the detergent will often have to be size reduced, classified or compounded with other materials to convert it to a marketable product. Laundry detergents are most often solid in the forms of powders and beads. Cleansers, such as scouring powders, are composed of very finely divided water insoluble powders, to minimize scratching of articles being scoured, and contain small quantities of water soluble detergents, which are also of small particle sizes. In the most widely used method of making commercial detergents the necessity of utilizing a separate size reduction operation after drying is obviated by spray drying, in which water is driven off from droplets of sprayed detergent composition, in solution-dispersion or slurry form, by the heat of hot gases in a spray tower through which the droplets fall. Thus, the end product, often a hollow sphere or similar shape of solid detergent composition is of approximately the desired size and, after removal of fines and oversize particles, may be directly employed as a detergent powder without the necessity for size reduction. However, such a drying operation requires crutching of the various detergent composition ingredients together in a liquid medium and sometimes this results in adverse chemical reactions between them. Furthermore, the high temperatures of the crutcher and the dryer can cause product degradation, especially in the spray dryer, if all the moisture has been driven off from the particles and the product is brought into contact with a hot gas or heated surface of the apparatus at a stagnant location or hot spot in it. Thus, it has been desirable to discover an economical method for readily removing moisture from detergent solutions without subjecting them to conditions that could cause decomposition of a heat-sensitive organic detergent.

Among evaporative methods that may be employed to dry detergents without degradation are vacuum drying techniques, such as flash vaporization and freeze drying. Although these do not cause thermal degradation of sensitive detergent they are comparatively expensive methods and still produce materials which must then be size reduced and blended with additional detergent composition materials. Of course, in such a blending differences in particle sizes and densities may later result in separations or sifting of product components. If homogenous particles are to be made from a crutcher mix the amount of moisture or other solvent to be evaporated from the mix may be excessive, making the mentioned expensive drying techniques impractical. Cheaper methods, such as tray drying at room temperatures, roll drying or ribbon drying, may be too slow or may be inapplicable to the detergents being treated. Even if useful, they will usually require an additional size reduction and/or blending operation.

The various disadvantages of the previously mentioned drying methods have been overcome in the present process, which is especially useful in the drying of heat sensitive detergents. The detergent solution, usually containing a substantial proportion of water, is deposited on finely divided particulate sorbent materials and the detergent, including water to be evaporated, is spread out over the surfaces of such material. Thus, the spreading action facilitates drying, since more active moistened surface is available for contact with a drying gas or for evaporation of the water into a vacuum, making it possible to employ lower drying temperatures and still obtain good drying rates. A further advantage is in the joining of the organic detergent to the sorbent material useful in those cases where the sorbent is a desired constituent of the final detergent composition or other composition in which the detergent is employed. Thus, the spreading or wetting action results in uniform distribution of the detergent throughout the product. The wetting action of the detergent promotes the spreading and with the detergent on the outer portion of the sorbent, its surface active and wetting effects can be exerted initially, when the product is used, thereby aiding in wetting or solubilizing the other detergent constituents.

The important concept of the invention is to spread the detergent solution so thinly over a finely divided base or sorbent that the drying medium, e.g., hot air, is in *contact* with the water and it is not necessary for the heat from the drying medium to travel through any appreciable thickness of sorbent or detergent solution, nor is it necessary for the water or water vapor to be removed to diffuse through an appreciable thickness of material to escape. Thus, when heat is applied evaporation of moisture may be almost instantaneous. Lower "hot air" temperatures may be used because of the better heat transfer and, due to the combined lower temperatures, evaporative cooling due to the quick heat transfer, and short heating periods, product degradation of heat sensitive materials is prevented.

In accordance with the present invention a method of drying a synthetic organic detergent comprises admixing said detergent, in solution in an aqueous medium, with a finely divided particulate solid sorptive material, suitable for incorporation in a detergent composition, to distribute the moisture in the detergent solution over the surfaces of the sorbent particles, and removing moisture from said particles at a temperature at which the detergent, which is also distributed over surfaces of the sorbent particles, will not be degraded. In preferred embodiments of the invention water constitutes a major proportion of the aqueous medium, the detergent is a higher alkyl benzene sulfonate, the particulate solid sorptive material is present in quantity greater than that of the solution of detergent salt, the mixing and drying operations take place in short periods of time and the drying is at a comparatively low temperature.

Various details, steps, operations, apparatuses employed, uses and advantages of the invention will be apparent from the description, taken in conjunction with the illustrative drawing of some embodiments thereof, in which drawing:

FIG. 1 is a schematic view of a high shear mixer in which a solution of detergent is distributed over particles of sorbent, a flash dryer in which moisture is removed from the detergent-sorbent-moisture particles and a cyclone separator in which particles are classified, after drying;

FIG. 2 is a schematic view of a similar high shear mixer in which both mixing and drying are effected, with separation of dried product from drying air being carried out in a cyclone separator, which also may serve to classify the product; and

FIG. 3 is a schematic view of a conventional ribbon mixer and spray means for coating sorbent therein with detergent solution, after which drying is effected in a vacuum dryer.

In FIG. 1, a high shear mixer **11**, of the horizontal cylindrical type, the main portion of which is commercially available from Littleford-Lodige Division of Littleford Bros. Inc., Cincinnati, Ohio, and is known, in unmodified condition, as a Lodige mixer, includes a horizontal cylindrical drum **13**, a pair of plows **15** and **17**, driven by main motor **19**, a pair of end scrapers **21** and **23**, also driven by main motor **19**, and a pair of choppers **25** and **27**, driven by individual chopper motors. Inlet passageway **29** is for the addition of sorbent to the mixer and passage **31**, communicating with distributing manifold **33** is for addition of detergent solution or suspension to the sorbent material.

In either a batch or continuous operation, the detergent coated sorbent containing moisture, so called detergent-sorbent-moisture particles, may be passed through exit line **35** to a flash evaporator **37**, held under vacuum. Moisture is drawn off as a vapor through line **39** and is liquefied in a condenser, not shown. Dried particulate product **41** may then be delivered through line **43** to a cyclone separator **45**, which may also act as a classifier. Any air or other gas present may be removed through line **47** with the product dropping out at **49**. By utilizing different exit ports for particulate material, classification may also be effected.

In FIG. 2 is shown a similar type of high shear modified Lodige mixer **51** in which drum **53** contains a pair of plows **55** and **57**, driven by main motor **59**, a pair of scrapers **61** and **63**, running off the same motor and a pair of choppers **65** and **67**, running off individual chopper motors. Passageway **69** is for addition of particulate solid material, such as sorbent, and passageway **71** and distributor **73** are for addition of detergent solution to be fairly evenly distributed over the particulate sorbent, which is vigorously moved and circulated by actions of the plows and choppers at the time of addition of the liquid detergent. A cloth sock vent **75** is provided to allow maintenance of the mixer at approximately atmospheric pressure or slightly above it, when materials and/or drying air are being added, while prevent losses of sorbent and detergent. It will be noted that moisture vapor can vent out through the walls of sock **75**. Drying air may be added to the mixer through **77** and diffuser or distributor **79** and, together with the actions of the choppers, plows and scrapers, serves to keep the particulate material in mixer **51** in rapid motion, while drying it. During such a drying operation or after it is completed, depending on whether a continuous or batch system is employed, the dried particles may be dropped to cyclone separator **80**, from which moisture vapor is removed through passage **81**, with product being taken off at **83**. Of course, classification can also be effected by selective removal of product from the cyclone.

FIG. 3 illustrates a screw or ribbon mixer **85**, of the open type, in which helical blade **87**, turning horizontally in trough **89** serves to keep a sorbent in motion under a spray manifold **91**, through which detergent solution is deposited on the sorbent particles. After completion of mixing, the sorbent-detergent-moisture particles are passed through outlet **93** to a dryer **95** operating under vacuum and at elevated temperature, although the temperature of the dryer is preferably always below that at which the detergent is unstable. It may be higher while the "wet" material is being dried but after removal of moisture should be below the instability point. Vacuum line **97** also serves as a means of removal of moisture vapor from the product. Dried product may be withdrawn after sufficient lowering of the moisture content, usually producing detergent-sorbent particles containing very little water.

In addition to the above described procedures, another preferred embodiment of the invention is a process carried out in accordance with FIG. 2 except for the absence of a separator, such as a cyclone or a Stokes settler. In such case the vent sock allows the drying air to be separated from the dried product. Thus, in such a process, whether effected batchwise or continuously, the high shear mixer and accessories for adding the various materials to it are the only means required.

In most preferred embodiments of the invention the mixing and drying operations are separate parts of a continuous operation to produce a dried product from detergent components plus an aqueous synthetic organic detergent solution.

Although the method is applicable to various detergents, including nonionic, cationic and anionic detergents, it is especially useful with respect to aqueous solutions of anionic detergent salts, which are often made by the neutralization of corresponding detergent acids in an aqueous medium. Among such salts those which it is preferred to employ are the higher alkyl benzene sulfonates, which today comprise a substantial proportion of the active synthetic organic detergent constituents of heavy duty laundry products, including detergent powders and scouring powders.

The preferred sulfonates are those wherein the higher alkyl is of 10 to 20 carbon atoms, preferably 10 to 18 carbon atoms and most preferably from 12 to 15 carbon atoms. Although branched chain alkyl benzene sulfonates are utilizable, the straight chain compounds, e.g., linear dodecyl benzene sulfonate and other linear higher alkyl benzene sulfonates, wherein the salt forming ion is an alkali metal, preferably sodium, are especially good, being readily dried and being free flowing after drying. Of the linear alkyl aryl sulfonates best are those wherein the alkyl is terminally attached to the benzene radical or is attached at the 2-carbon. Of course, these compounds can be employed in mixture with other anionic or nonionic detergents or such other detergents may be used in replacement of the alkyl benzene sulfonates. Thus, higher alkyl sulfates, higher alkyl sulfonates, higher acyl sarcosides, higher acyl Nmethyl taurides, higher alkyl lower alkoxy sulfates, higher alkyl poly-lower alkoxy sulfates, middle alkyl phenoxy ethoxy ethanol sulfates and many other of the well known anionic detergent salts, preferably the alkali metal salts, may be used. "Higher" means of 10 to 20 carbon atoms, "middle" of 7 to 9 and "lower" of one to six carbons. Linear alkyls are usually preferred. Among the useful nonionic detergents employed are the block copolymers of ethylene oxide and propylene oxide of high molecular weight (Pluronics), higher alkyl ethers of poly-lower alkoxy alkanols, higher alkyl phenoxy polyethoxy ethanols, higher fatty acid esters of poly-lower alkoxy alkanols and similar well known detergents. The cationic detergent materials include cetyl trimethyl ammonium bromide, dioctyl dimethyl ammonium chloride, pyridinium halides, benzethonium chloride and similar quaternary and corresponding phosphonium compounds. In addition, amphoteric and ampholytic detergents any also be employed, either alone or in mixture with others with which they are compatible. Generally, mixtures of anionic and cationic detergents should not be used. In all the preceding cases mentioned, it is preferred that the detergent be one which is normally solid, so that the sorbent-detergent particles made will be most readily free flowing. However, liquid detergents may be employed so long as the proportions thereof are kept within limits to produce solid and free flowing particulate products. The solution of detergent material may include with it various impurities, byproducts of previous chemical reactions and solvents, in addition to water. Thus, free oil or unreacted starting materials or derivatives thereof, acids, inorganic salts, e.g., sodium sulfate, and alcohols, e.g., ethanol may be present, generally to a limited extent, usually less than 20 percent and most frequently less than **10%** of the detergent solution. If desired, adjuvants for the final product may be included with the detergent solution. Such adjuvants will be discussed in more detail later.

The sorbent or particulate substrate onto which the detergent solution is deposited may be any suitable material, over the surface of which the water in the detergent solution will spread to make it more readily vaporizable. Thus, it is not necessary that the substrate be one which is water soluble or permeable and it will often be desirable for the water in the detergent solution to fail to penetrate deeply into the particles. In addition to the properties of the substrate or sorbent itself, other factors are controllable to regulate penetration, including proportions of detergent solution and substrate, concentration of detergent in the solution, viscosity of the solution, speed of mixing, particle sizes of sorbents and sprayed droplets of detergent solution, vigorousness of shearing action and speed of drying, if drying is effected in the mixer. While various sorbents may be employed, it is preferred to use those which perform a useful function in the desired ultimate product in which the detergent is incorporated. Thus, in preferred embodiments of the invention the sorbent may be a builder or a filler salt, a scouring material, adjuvant material or mixtures thereof. Among the useful adjuvants may be mentioned slip-promoting agents, e.g., talc, magnesium silicate; bulk increasing materials, e.g., alpha cellulose, asbestos; and anti-redeposition agents, e.g., polyvinyl alcohol, sodium carboxymethyl cellulose. Primarily, however, the sorbent is intended to be a builder or filler salt, such as trisodium phosphate, pentasodium tripolyphosphate, tetrasodium pyrophosphate, sodium carbonate, sodium bicarbonate, borax, boric acid (convertible to borates at alkaline pH's), sodium sulfate, sodium bisulfate, disodium phosphate, monosodium phosphate or other corresponding alkali metal, e.g., potassium, ammonium or other water soluble salts. Among preferred substrates, of the insoluble type, the most important is silex, a powdered silica. However, other such materials include talc, calcium silicate, pumice, tricalcium phosphate and other known scouring and polishing agents, usually in finely divided powder form. All such materials will be referred to herein as sorbents.

Adjuvants that may be employed, either as solids with the sorbent or dissolved in the detergent solution, as may be most suitable, include builder-sequestrants such as nitrilotriacetic acid and ethylene diamine tetraacetic acid; bleaches, e.g., trichloroisocyanuric acid; stain removing chemicals, e.g., enzymes; oxidizing agents, e.g., sodium perborate; reducing agents, e.g., sodium bromide; bulking materials; pH regulators; brighteners; foaming agents; fungicides; bactericides; perfumes; colorants; viscosity enhancing compounds; and soil-suspending and anti-redeposition agents. The adjuvants will usually comprise a minor proportion of the final dried product, generally being less than 5 percent thereof each and preferably being less than 2 percent each, with a total adjuvant content of less than 20 percent, preferably less than 10 percent. If nitrilotriacetic acid, ethylene diamine tetraacetic acid, salt thereof or other organic material is used as a principal builder the proportion may be increased to that for inorganic builder salts. The above remarks with respect to NTA are with respect to the detersive or builder effects thereof, which are satisfactory. However, unless clearance is received for the use of NTA in such products, it will be omitted. The trichloroisocyanuric acid, being unstable in solution, will usually be employed as a powder. As with other materials which are not stable to water, it may be added subsequent to drying.

The detergent proportion in the final dried product will generally be from 2 to 30 percent thereof, preferably from 5 to 20 percent thereof. The weight of sorbent, whether water soluble builder or filler salt or mixture, or inorganic scouring material or mixer, or other insoluble material, will usually be from one or two to 40 times the weight of the detergent, e.g., alkyl benzene sulfonate, and preferably is from six to 14 times the weight thereof. Thus, the proportion of such material in the final product will usually be from 50 to 95 percent thereof, preferably from 60 to 85 percent. It will generally be desirable to have the final moisture content comparatively low. Usually, this will be from 0.1 to 10 percent and preferably will be under 5 percent, with a preferred range being from about 0.1 to 2 percent.

The moisture content of the sorbent before deposition thereof the aqueous solution of detergent will normally be as low as feasible so that best spreading action will be obtained, with fastest drying resulting, too. Anhydrous salts or scouring or polishing agents are much preferred but small amounts of moisture, up to 10 percent, if strongly held in hydrate form, preferably no more than 5 percent and most preferably of 1 or 2 percent may be present. The solution of detergent, which may include some undissolved detergent also, will usually have from 10 to 75 percent of detergent present, the balance being an aqueous solvent medium. Preferably, as in the case of the alkyl benzene sulfonates, detergent content will be from 30 to 65 percent thereof. Usually it will be best for the drying operation to have the solids content of the detergent solution as high as feasible under the circumstances. For linear higher alkyl benzene sulfonates such content will most preferably be from 50 to 60 percent. The balance of the solution, the aqueous medium, will normally be from 80 to 100 percent of water, with some solvent or adjuvants also present, when desired, or when their presence is dictated by processing considerations.

The particle sizes of the sorbent will generally be small enough so that most or substantially all of the substrate will pass a No. 40 U.S. sieve. For ultimate scouring cleanser use, most or substantially all, over 99 percent, of the particles pass through a No. 100 sieve and a major proportion thereof will pass through a No. 200 sieve. For heavy duty detergent powder use, it is preferred that substantially all of the particles fail to pass through a No. 200 sieve and it is most preferred that a major proportion thereof should be large enough so as not to pass through a No. 60 sieve.

Particle size of the sorbent is a factor in determining the end use properties of the cleaning products mentioned but the diameter of the solution droplets deposited on the solid particles is of lesser importance. Thus, when mixing is rapid and the sorbent is in a state of high turbulence the droplets may be broken into smaller particles before contacting the sorbent. Therefore, in many cases even streams of detergent solution may be utilized, since these are rapidly broken into small droplets and the chopping action of the mixer, if a modified Lodige mixer is employed, further serves to distribute the liquid over the substrates. Nevertheless, it is generally preferable to maintain the droplet size less than 0.050 cm. and preferably it will be from 0.005 cm. to 0.02 cm. Of course, the point of addition of the detergent solution and its direction should be such as to make certain that it will impinge on the sorbent and not hit a wall surface or be swept out through a vent or other outlet before it has an opportunity to make such contact.

The mixer in which the sorbent is continuously moved while the detergent solution is being deposited on the particles thereof is an efficient device for moving virtually all the particles thereof all the time, without the existence of dead spaces. Preferably, the particle movement is irregular, whereby it is meant that a particular particle does not trace the same course repeatedly. Although particle velocity is dependent to a large extent upon mixer design, the particles will usually be moving at speeds of about 50 to 5,000 feet per minute. To assist in continually changing the "bed" or whirling mass of substrate particles, the mixer will normally be no more than three-fourths full of the sorbent and preferably will be from one-fourth to one-half full. The pressure in the mixer will normally be about atmospheric, when the apparatus is not connected to vacuum drying means. Thus, it will usually be from 0 to 5 lbs. per sq. in gauge, preferably from 0.1 to 1 p.s.i.g. However, if vacuum drying is employed in the mixer the vacuum may cause the pressure to be from 0.5 to 14 p.s.i. absolute. The temperature in the mixer may be from 10°C. to 110°C. and even higher, up to the degradation point of the detergent or other material present, if desired. Whether the temperature can be maintained much above 110°C., e.g., up to 250°C., after removal of water will depend on the length of time to which the detergent or other sensitive material is exposed to it in the mixer and on the heat sensitivity of such material. Normally, temperatures in the mixer will rarely exceed 150°C. and most often will be from 20°C. to 70°C.

The speed of mixing will preferably be as high as feasible and time of mixing or addition of detergent solution will preferably be as short as possible. Such conditions are obtainable, with good mixing and/or drying of the detergent solution in the mixer when a high shear mixer of the Lodige type is employed, equipped with an air supply or attached to vacuum means to effect drying with mixing. The excellent mixing and contacting actions obtained in such a high shear mixer are apparently due to the combination of ploughing and scraping actions, at moderate speeds, about 150 revolutions per minute, or 700 to 1,100 ft. per minute plow tip speed, combined with higher speed chopping actions, wherein the chopper blades rotate at about 3,600 r.p.m. Under such circumstances, the addition and mixing operation may take as little as 5 to 10 seconds, although in other mixers this time may be increased to up to about 5 minutes. If drying means are employed in the high shear mixer, the drying can be effected in from 2 seconds to 2 minutes. Even less than 2 seconds can result in good drying, as when vacuum and comparatively high temperatures are both employed. Under ideal circumstances, such as those obtained in a flash dryer, drying can be effected in as little as 0.01 second, essentially instantaneously. Preferably, drying takes less than 10 seconds.

If a gas is used to aid in drying and circulating the sorbent and detergent solution in the mixer, it will usually be at a temperature in the range of 10° to 150°C., preferably from 20° to 70°C., and the gas will preferably be air, although other inert gases, e.g., nitrogen, carbon dioxide, halocarbons may also be used. Such gases may also be used when drying is effected in an additional drying apparatus and not in the mixer itself. The rate of air flow through the mixer may be that resulting from blowing the air or other gas through a pipe of one-eighth to 2 inches inside diameter, at a pressure from 5 to 200 lbs. per sq. inch gauge. The air may be diffused or may be blown into the mixer directly from the pipe. The flow rate of the air, which is ventable through a permeable venting sock, can be such as to result in a change of air in the mixer every 1 to 50 seconds.

Under the conditions previously described, effective drying of detergent solutions can be obtained quickly and efficiently, with additional processing benefits which improve the "homogeneity" of the product. The processes described herein and illustrated in the drawing can be carried out continuously which is preferable, or batchwise, if desired. The selection of the type of process may depend on the characteristics of the detergent and the composition in which it is to be incorporated. One may combine or separate the addition, mixing and drying operations and by control of the mixing technique a certain desired amount of agglomeration or size reduction may also be obtained. Under some circumstances, as when a fragile material is being treated which must be handled gently, a plurality of particularly suitable apparatuses may preferably be used for effecting the various processing operations.

As illustrated in FIG. 1, drying is not effected in the Lodige high shear mixer but that piece of equipment is used only to distribute the detergent solution over the sorbent particles. Subsequently, the material is flash dried, usually at an absolute pressure of from 0.5 to 14 p.s.i., with the particulate material being dried being initially at a temperature of from about 10° to 110°C. If heating is desired, the particulate sorbent may often be heated beforehand and the detergent solution, at room temperature or slightly elevated temperature, may be sprayed onto it. In such cases, means may be provided in the mixer for removing any water vapor generated upon contact of the solution and sorbent particles. In place of the flash vaporizer, other drying means may also be used, including tray dryers, fluidized beds with drying gas permeating them, etc. Then, if desired, as illustrated in FIG. 1, a cyclone separator may be employed to classify the particles to the desired size range and to remove any entrained gases from them. In essence, the operation illustrated in FIG. 2 is the same as that of FIG. 1 with the exception that the drying is effected in the mixer. Such an operation is best suited for use with an inert sorbent, such as polishing material intended for incorporation in scouring cleansers. The larger surface area promotes more rapid drying and the particles are not as susceptible to size reduction or fragmentation disadvantages as those intended for use in detergent powders. On the other hand, the apparatus of FIG. 3, utilizing a conventional ribbon blender or corresponding mixer, is particularly useful with sensitive materials which cannot stand either high temperature or high shear actions.

The advantages of the present methods have been referred to previously and will not be reiterated at length here. However, although after reading of this description it may appear that the comparatively simple processes described could have been obvious to one of skill in the art confronted with the problem of making detergent compositions from aqueous detergent solutions without the use of high temperatures or crutching operations, there were many reasons why these methods were not previously practiced. For example, synthetic organic detergents are generally somewhat hygroscopic. They tend to absorb moisture and become lumpy or tacky. When such detergents are dried from solutions, they tend to become gelatinous or pasty and the colloidal product made is difficult to dry thoroughly. Yet, with the use of a high shear mixer and a sorbent, it is found that the wetting action of the detergent apparently assists in distributing water over the sorbent to such an extent that gummy dried product is not obtained. Also, the mass transfer rate of water from the initial droplets applied to the substrate is greater than the heat transfer rate so that there is not formed on the substrate a blocking deposit of detergent which could prevent further distribution of moisture over the sorbent particles. Therefore, instead of requiring more time and higher temperature for drying, less time and lower temperatures suffice. The invention possesses another significant advantage in that it tends to hold the detergent tightly to the substrate material and to join other components of the final product to the substrate. Thus, a more uniform detergent composition is produced and it may be of a higher bulk density than comparable spray dried products, often being from 0.5 to 1 g./ml. rather than from 0.2 to 0.4 g./ml. Yet, if dried detergent, without substrate, were to be desired, one could employ a sorbent to aid in the drying process and then could separate the sorbent from the detergent by mechanical means, including subjection to repeated shocks, followed by centrifugal action. Hydratable builder salts may be employed and will give up water of hydration during the present drying processes. Fast drying is obtainable without the need for utilizing dangerously high temperatures of drying gas. Finally, detergent composition ingredients may suffice as the sorbents, carriers or bases to be employed.

The following examples illustrate various preferred embodiments of the invention. Unless otherwise indicated all parts are by weight and all temperatures are in degrees centigrade.

EXAMPLE 1

Using the equipment illustrated in FIG. 1, a high shear Lodige-type mixer having a volume of approximately 12 cubic feet and equipped with plows and scrapers turning at tip speeds of about 900 ft. per. min. and, chopper blades moving at about 3,600 r.p.m. (5,000 ft. per. min.) there are added 76.8 parts of finely ground alpha quartz (silex), a major proportion of which passes a No. 200 sieve and substantially all of which passes through a No. 100 U.S. sieve. To the quartz scouring material are added 8.2 parts of anhydrous trisodium phosphate, also finely divided so that substantially all of it passes a No. 100 sieve, with a major proportion passing through a No. 200 sieve. The two sorbents (they may be adsorbents or absorbents) are blended together for approximately one-half minute, after which the dispersion is uniform. Then, about eight parts of an aqueous solution of sodium linear dodecyl benzene sulfonate detergent in which some insoluble detergent is also present is pumped into the mixer and is sprayed onto the moving sorbent mixture. The alkyl aryl sulfonate is 56.5 percent solids and 87.5 percent of that is pure sodium dodecyl benzene sulfonate. The solids include 1.6 percent of oil (ether solubles), 5 percent sodium sulfate, 7 percent ethanol and the balance is water. It is sprayed onto the surface of the moving sorbent so that the droplets applied to the sorbent are about 0.01 cm. in average diameter. The addition of the detergent slurry or solution takes place over a period of 1 minute and after that, mixing is continued for an additional 1 ½ minutes. It is considered that after that time the moisture in the detergent is fairly evenly distributed over the particles of silex and inorganic builder salt.

After completion of mixing, the detergent-sorbent-moisture particles are discharged to a flash dryer, in which moisture is removed from them in a flashing operation at a pressure 2 p.s.i.a., the moisture being removed within 10 seconds at room temperature, 25°C., to reduce the product to a substantially anhydrous state, containing 0.2 percent moisture. The particle sizes of the product resulting are slightly larger than those of the substrates charged but generally within the same particle size specifications. The detergent is held tightly to the individual particles of sorbent, despite the removal of the moisture. Subsequently, the materials are passed to a cyclone separator, where by control of the removal port locations and/or the velocity of the stream of materials, any product coarser than about 100 mesh is removed. The resulting material, with adjuvants, is useful as a foaming scouring cleanser. On storage, it does not cake or lump and even in the presence of various adjuvants which are unstable when in contact with synthetic organic detergent, is of satisfactory stability.

In other experiments, instead of blending in the detergent solution with the moving substrate, it is added on top of a stationary bed of substrate and mixing is commenced after addition. Although the product resulting is useful, it is not as readily dried and is not as uniform as that made by the previously exemplified method. In other variations of the principal method small percentages of various adjuvants are included in the sorbent mixture. Thus, 0.5 percent of hexachlorophene, 1 percent of sodium trichlorocyanurate, 0.3 perfume and 0.05 percent coloring pigment are included with the sorbent. This material dries equally well and although there appears to be some loss of perfume, the product still has the distinct bouquet of the perfume employed. Also, when such a composition is made in the Lodige mixer illustrated in FIG. **2**, wherein drying takes place in the mixer in the presence of drying air at 70°C. circulating through the mixer at the rate of **10** air changes per minute with the mixer 40 percent full of sorbent, drying is effected to a moisture content of 0.3 percent within a period of 1 minute. Product characteristics obtained are the same as those previously described in this example and the classifying action of the cyclone separator may or may not be utilized. In either case, the result is an excellent scouring cleanser cheaply and rapidly made with economical and efficient processing equipment.

EXAMPLE 2

	Percent
Pentasodium tripolyphosphate	40
Boric acid (anhydrous)	5
Sodium sulfate (anhydrous)	35
Optical brighteners	0.5
Stain removing enzymes	2.0
Sodium carboxymethyl cellulose	0.5
Polyvinyl alcohol	0.5
Sodium n-tetradecyl benzene sulfonate (50% solution-suspension in water)	16.5

The above built synthetic organic detergent composition is made by adding the sodium n-tetradecyl benzene sulfonate solution to the moving mixture of the other materials, as a sorbent bed. Drying is effected with hot air being blown through the Lodige mixer during the period of addition and shortly after, for a total of 1 minute. The hydratable salts initially pick up the moisture and then it is evaporated from them due to the intimate mixing of the hot air and the finely divided particles, which are within the size range of 40 to 100 mesh. Moisture content is reduced to 0.2 to 0.4 percent. The product resulting, which may be cyclone separated from the gas and fines which may be present, is useful as a heavy duty built synthetic detergent.

Variations of the formula are also made, wherein the builders are changed, using a major proportion of sodium nitrilotriacetic acid, sodium salt, instead of the pentasodium tripolyphosphate. Also, different adjuvants may be used and similar results are obtained. In other experiments, instead of batch operations, materials are continuously added and removed and drying is satisfactorily effected with the making of an acceptable product. In some cases, when moisture contents are not sufficiently low after removal from the high shear mixer or from a ribbon or Day mixer, additional drying is effected in a tray dryer at comparatively low temperature, e.g., 50°C. or under vacuum, e.g., 5 p.s.i.a.

After removal of the dried detergent a check on bulk density indicates that the material is heavier than the normal spray dried detergents, having a bulk density of about 0.6 g./ml. This is advantageous in marketing a more compact product, with the consequent savings of storage and shipment costs. Such product is readily soluble in wash water, at a rate comparable to that of spray dried detergent beads.

EXAMPLE 3

	Parts
Magnesium linear alkyl benzene sulfonate slurry (55% solids; 88.5% of the solids content being MLABS)	7.5
Trisodium phosphate	3.4
Calcium silicate (synthetic)	1.0
Sodium carbonate	10.0
Calcium carbonate	77.6
Perfume, coloring	0.5

EXAMPLE 4

Sodium linear alkyl benzene sulfonate slurry (56.5% solids; 87.5% of the solids being SLABS)	5.1
Sodium bromide	0.6
Boric acid, anhydrous	2.0
Sodium carbonate, anhydrous	4.0
Sodium sulfate, anhydrous	88.3

EXAMPLE 5

	Parts
Magnesium linear alkyl benzene sulfonate slurry (55% solids contents; 88.5% of the solids being MLABS)	16.0
Trisodium phosphate, anhydrous	8.4
Sodium bromide	0.6
Calcium silicate	1.5
Sodium carbonate	4.0
Alpha quartz (silex)	69.2
Optical brightening compounds	0.3

In the preceding three examples the linear alkyl group is of an average of about 13 carbon atoms and includes C_{12}, C_{13}, C_{14} and C_{15} alkyls. The various materials described are processed in the mixer of FIG. **1** according to the method of Example 1, with the conditions of addition, particle sizes, etc., being essentially the same as in that example. In all cases the formula constituents, except for the aqueous slurries, are blended together and the slurries are added to them over a period or about 1 to 2 minutes, after which mixing is continued for an additional 1 to 2 minutes and the mixtures are sent to a vacuum or flash dryer, in which almost all of the moisture is removed, so that the final product has a moisture content of less than 1 percent. The products of Examples 3 and 5 are suitable for use as scouring cleansers and that of Example 4 may be employed as a phosphate-free heavy duty synthetic detergent or may be added to insoluble polishing materials,e.g.,calcium silicate, calcium carbonate or alpha quartz, to produce scouring cleansers. During the mixing in of the slurry of synthetic detergent with the other dry constituents, the surface thickness of the water layer on the particles produced is less than 0.01 cm. and often is less than 0.005 cm., which contributes to the quick drying of the particles in the flash evaporator. It is notable that the moisture is on the exterior of the particles, from which it is easily removed, rather than on the interior surrounded by insulating material.

In similar experiments, the same formulations are made according to the method of Example 2, utilizing the equipment of FIG. 3. Although the product obtained is acceptable, it is not as good as that made by the method of Example 1, whether utilizing the apparatus of FIG. 1 or that of FIG. 2.

Modifications of the formulation are employed, substituting 50 percent solids content slurries of 90 percent active ingredient detergent materials of the monoglyceride sulfate, tallow alcohol sulfate, lauryl polyoxyethylene sulfate, olefin sulfonate and dioctyl sulfosuccinate types, as their sodium or potassium salts. The products resulting are useful as detergents for cleaning textiles or hard surfaces, depending on the contents of insoluble polishing materials. In all such operations, drying is effected quickly and efficiently, without thermal degradation of the detergent or other ingredients. In a similar manner, others of the mentioned soluble and insoluble inorganic salts and compounds may be substituted for those of the above formulas and similar results are obtainable. Also, adjuvants may be incorporated in the final product after drying. Such procedures are often followed when such adjuvants, e.g., bleaches, perfumes, are sensitive to the water present in the detergent slurry.

The invention has been described with respect to various examples thereof. It is evident that it is not to be considered as limited thereby since these examples are only intended to be illustrative.

What is claimed is:

1. A method of drying a heat-sensitive synthetic organic detergent which comprises admixing said detergent, in solution in an aqueous medium, with a particulate solid sorptive material, suitable for incorporation in a detergent composition, to distribute the moisture in the detergent solution over the surfaces of the sorbent particles, and removing moisture from said particles at a temperature at which the detergent, which is also distributed over surfaces of the sorbent particles, will not be degraded.

2. A method according to claim 1 wherein the synthetic organic detergent is an anionic detergent salt, the aqueous medium, exclusive of detergent, comprises a major proportion of water and the particulate solid sorptive material is present in quantity greater than that of the solution of detergent salt.

3. A method according to claim 2 wherein the synthetic organic anionic detergent salt is an alkali metal higher alkyl benzene sulfonate, the aqueous medium is from 80 to 100 percent water, the alkyl benzene sulfonate comprises from 10 to 75 percent of the total weight of such detergent salt and aqueous medium in which it is dissolved, the particulate solid sorptive material present comprises from two to 40 times the weight of the alkyl benzene sulfonate, the mixing operation takes from 5 seconds to 5 minutes and the drying operation takes from 0.01 second to 2 minutes.

4. A method according to claim 3 wherein the alkali metal higher alkyl benzene sulfonate is a sodium higher alkyl benzene sulfonate wherein the alkyl is a linear alkyl of 12 to 15 carbon atoms, the solution of said alkyl benzene sulfonate comprises from 30 to 65 percent of said sulfonate, and the particulate solid sorptive material present is from six to 14 times the weight of the alkyl benzene sulfonate.

5. A method according to claim 4 wherein the sorptive material is selected from the group consisting of silica, trisodium phosphate, pentasodium tripolyphosphate, boric acid and sodium sulfate.

6. A method according to claim 5 wherein the admixing of detergent and particulate sorptive material takes place in a closed high shear mixer in which the mixture is in continuous highly turbulent motion and the drying is effected by hot gas forced into the mixer during at least a portion of the operation thereof.

7. A method according to claim 6 wherein the gas is air and after drying is effected, the air containing evaporated moisture from the detergent-sorbent-moisture particles is separated from the dried detergent-sorbent particles.

8. A method according to claim 7 wherein the separation is effected in a cyclone separator.

9. A method according to claim 5 wherein the moisture is removed from the detergent-sorbent-moisture particles by flash drying.

10. A method according to claim 5 wherein the surface temperature of the detergent-sorbent-moisture particles during the drying operation is from about 10° to 110°C.

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