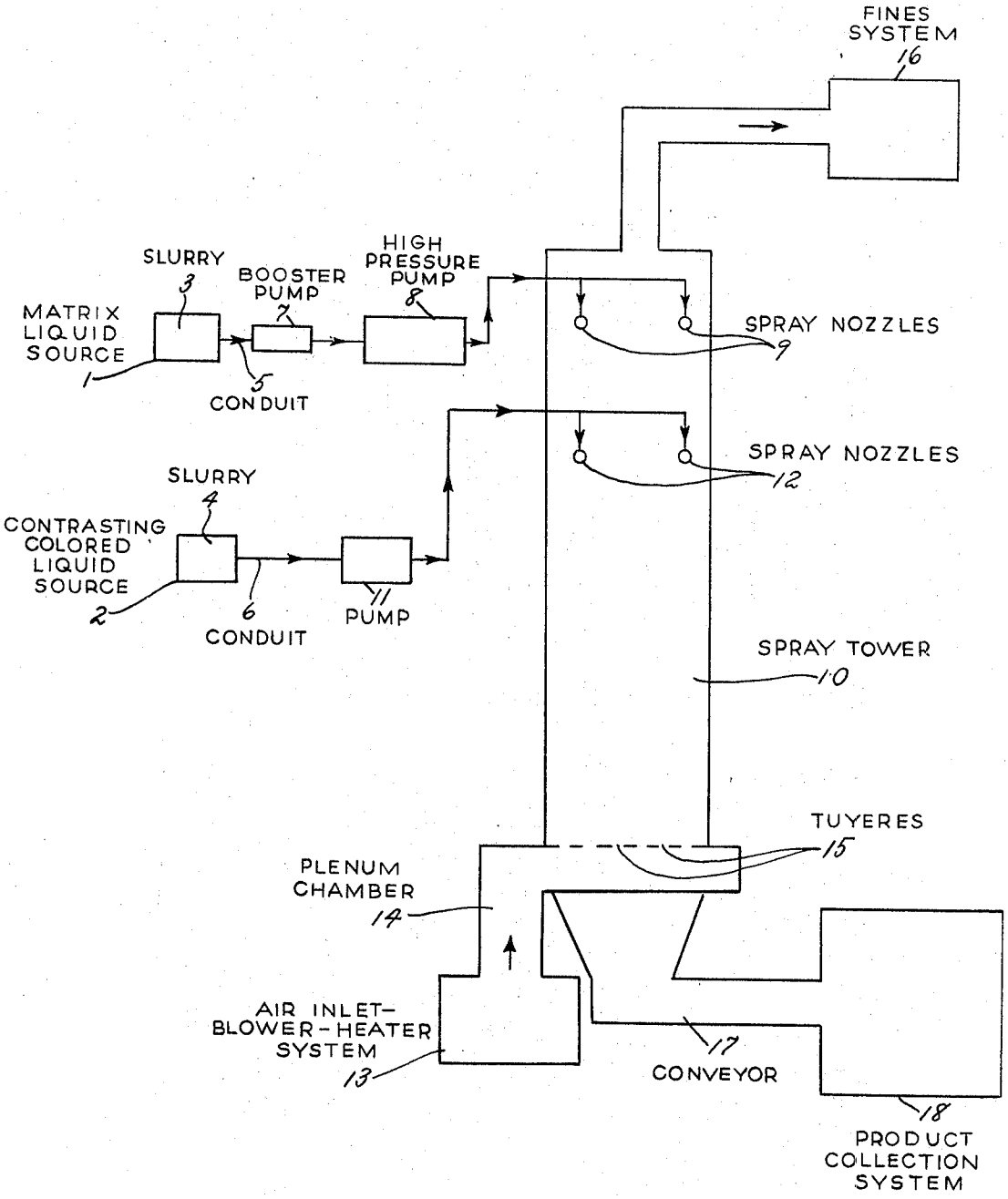
July 7, 1970 J. A. CAVATAIO ET AL 3,519,054

PROCESS FOR PRODUCING A PARTICULATE PRODUCTS

Filed Jan. 6, 1969



INVENTORS
JERALD ALBERT CAVATAIO
JOHN ALEXANDER MONICK

BY Saul R. Bard

ATTORNEY

United States Patent Office 3,519,054 Patented July 7, 1970

3,519,054

PROCESS FOR PRODUCING A PARTICULATE PRODUCT

Jerald Albert Cavataio, Far Hills, and John Alexander Monick, Teaneck, N.J., assignors to Colgate-Palmolive Company, New York, N.Y., a corporation of Delaware

Continuation-in-part of abandoned application Ser. No. 654,423, July 19, 1967. This application Jan. 6, 1969, Ser. No. 830,555

Int. Cl. B01d 1/18

U.S. Cl. 159—48 8 Claims

ABSTRACT OF THE DISCLOSURE

Process comprising spraying two liquid streams downwardly in the form of droplets into a spray tower, one stream being sprayed from a level 15 to 60% below that of the other, into an upwardly flowing stream of drying gas whereby the droplets are converted into dried multi-colored particles.

This invention relates to a continuous process for the production of a particulate product and, more particularly, to a continuous process for the production of a particulate product by spray drying. This application is a continuation-in-part of U.S. application Ser. No. 654,423 filed July 19, 1967, now abandoned.

The production of particulate products such as detergent compositions containing beads or similar particles by spray drying liquids from nozzles located on a ring manifold placed in the upper portion of a spray tower is known. The liquids are sprayed in a plurality of droplets which are dried to solid particles by the action of a drying gas passing through the spray tower. Two systems of drying can be used, either the concurrent system wherein the drying gas passes in the same direction or concurrently with the direction of the spray or the countercurrent system where the drying gas passes in an opposite direction or countercurrently to the direction of the spray. Each system has its own advantages and, particularly in the production of spray dried detergents, the countercurrent system has been found to be more advantageous than the concurrent system in that less fines are produced, there is greater heat economy, and the product is more granular and less friable.

Where it is desired to prepare particulate products such as typified by multi-colored detergents, additional considerations have to be taken into account. The purpose of multi-color is to provide a consumer product which is pleasing to the eye. Therefore, the different colored particles have to be distinctive, one color preferably furnishing a background for the other. In order to provide a distinctive mixture and yet adhere to economical and simple procedures, the background material known as the matrix is generally present in high proportion and in its natural color, which merely means that no colorant has been added. In the case of detergents the natural color is usually white and this color provides an excellent background although light blue has also been found to be pleasing to the eye. The colored material is generally the same as the matrix composition-wise, although it can be different, with colorant added. It is usually present in a much smaller proportion and is of a color which contrasts sharply with that of the matrix material. For example, deep blues stand out very well, especially against a background of white or light blue.

Taking the foregoing into account, an attempt was made to prepare a multi-colored particulate product using a conventional countercurrent spray drying system and materials having sharply contrasting colors, the objective being, among others, to obtain a product wherein one

color was sharply distinctive from the color of the background material. It was found that the particles which were to contrast with the matrix did not stand out as expected. Instead, they had a "dusty" appearance and the mixture was not pleasing to the eye. Close examination of these particles revealed that the "dusty" coating on the particles were composed of fines, which are particles passing through 100 mesh. These fines were made up of background or matrix material.

To overcome the problem of the fines, it was proposed to install a more sophisticated fines removal system, but the high cost of removing the small amount of fines with such as system is prohibitive. In this respect, it should be noted that a large percentage of fines arise from very small droplets which are formed at the spray nozzle. Although the size of most of the droplets can be kept within certain ranges by adjusting the pressure and the size of the nozzle orifice, small numbers of very small droplets cannot be prevented. Other fines are produced by the collision of particles with one another and with the walls of the spray tower. Since the interplay of currents of air, for example, in the form of eddies, tower temperatures, and gravity cause the fines to move in an unpredictable manner in the spray tower, anything short of separate spray drying of matrix and colored particles appeared to be impractical and uneconomical.

In addition to the problem of fines resulting from countercurrent spray drying of a multi-colored particulate product, there is a further problem of agglomeration especially when one or more matrix particles and colored particles join together. The result in this case is similar to that of the "dusty" coating of fines described above in that the colored particles lose their distinctiveness. Problems of the aforedescribed nature are likewise manifest in connection with countercurrent spray-drying techniques having as an object the provision of particulate products whether uni-colored or otherwise devoid of fines, agglomerates and the like.

It is, therefore, an object of the present invention to provide a process for the production of particulate products wherein the foregoing problems are eliminated or at least mitigated to a substantial degree.

A further object is to provide a process for the preparation of particulate products whereby the product is comprised of discrete particles substantially free from agglomeration.

Another object is to provide a process for the preparation of a multi-colored particulate product wherein fines are prevented from adhering to the surface of particles having a different color than the fines.

Other objects and advantages will be apparent from the following description.

In accordance with the invention, a process has been found for the continuous production of a particulate product in a countercurrent spray drying system comprising the following steps:

(1) Forming a first stream of a liquid matrix capable of being converted into solid particles by spray drying;

(2) Passing the first stream to a predetermined point of entry in the upper portion of a spray tower;

(3) Forming a second stream of liquid and capable of being converted into solid particles by spray drying;

(4) Passing the second stream to a point of entry in the spray tower at a level below that of the point of entry of the first stream at which substantially all of the fines formed at the point of entry of said first stream have been substantially dried and at a level above the bottom of the spray tower sufficient to substantially dry droplets formed by the second stream;

(5) Passing a stream of drying gas upwardly through the tower;

(6) Simultaneously spraying the two streams from their respective points of entry downwardly in the form of a plurality of liquid droplets into the upwardly flowing stream of drying gas, whereby the droplets are dried and converted to solid particles; and

(7) Removing the dried product containing a homogeneous mixture of substantially discrete particles from the drying gas. In those instances where multi-colored products are being prepared, each of the respective feed streams will be of a constant color, i.e., easily differentiated visually.

The spray tower apparatus which can be used to carry out the above-described process comprises:

(1) A vertical spray tower;

(2) Means for providing a first liquid capable of being converted into particles by spray drying;

(3) Means for providing a second liquid capable of being converted into particles by spray drying;

(4) Means for feeding the first liquid to a predetermined point of entry in the upper portion of the tower;

(5) Means for feeding the second liquid to a point of entry about 15% to 60% below the level of the point of entry of the first liquid, the percentage based on the distance from the bottom of the spray tower to the point of entry of the first liquid;

(6) Means for spraying each of the liquids from their respective points of entry into the tower in such a manner so as to form a plurality of liquid droplets;

(7) Means for passing a stream of drying gas countercurrently through said tower to dry the droplets and convert the droplets to particles; and

(8) Means for removing the dried particles from the tower.

The above process when utilized for the production of multi-colored particulate product provides particles of one color substantially distinctive from the particles of another color; the fines are substantially prevented from adhering to the surface of particles having a different color than the fines; and the particles are discrete being substantially free from agglomeration.

It is surprising that the adherence of fines and agglomeration are minimized in view of the fact that the fines from the matrix pass along the same path as they do when one spray level is used and the contrasting colored droplets from the lower level are exposed to the fines when such colored droplets are in a wet, tacky state as they are converted to dry particles.

A preferred and unique feature of this invention is the use of a fine spray at the upper level to provide fine particles (first stream) and a relatively coarse spray at the lower level to provide relatively coarse particles (second stream). The different sprays are generally obtained by adjusting the pressure and nozzle sizes, which are more fully described below. The sprays can be defined in terms of the size of the particles which result from the droplets formed by the first stream being of a size sufficient to provide particles of −20 to +60 mesh in major proportion and the droplets formed by the second stream being of a size sufficient to provide particles of −8 to +20 mesh in major proportion. Using this feature, it was unexpectedly found that the larger particles do not attract fines because of their size and are even more distinctive colorwise than when the matrix is a similar size to that of the contrasting colored particles.

Referring to the drawing:

The sole figure is a diagrammatic side view showing a conventional spray tower system or apparatus modified to carry out the inventive process.

An example of a spray tower apparatus which can be subjected to modification for use in this invention is disclosed in U.S. Pat. 2,851,097 issued to Ledgett on Sept. 9, 1958. This patent discloses apparatus for drying a product continuously supplied under pressure into a ring shaped manifold with a series of discharge nozzles extending into the tower interior. The tower of the Ledgett patent and the description provided therein can be used to supply any details of the spray system which are incidental to subject inventive process and apparatus.

The following detailed description of the inventive process and apparatus with reference to the drawing is set forth in terms of a preferred embodiment of this invention, i.e., the production of a multi-colored, particulate detergent product, although any liquid materials which are capable of being converted to particles on spray drying i.e., uni-colored, multi-colored etc. are contemplated for use herein. In this same vein, even though all of the various contrasting colors are contemplated, the matrix is best exemplified by an uncolored or light blue liquid detergent composition which can be spray dried, the matrix being defined as the base or background material which is present in major proportion in the composition. The liquid matrix is said to be uncolored when no color is added to it during processing. Generally, detergent compositions, which have not been colored are white and so provide a good background for a mixture of particles with a distinctive appearance. As a general rule, white or light blue matrices are used in multi-colored detergent compositions, not only because they provide a good background, but for reasons of facility and economy. The colored liquid used in minor proportion can be exemplified by a liquid whose color provides a sharp contrast to the white or light blue background. Dark hues of blue are particularly desirable. Generally, both the matrix and the contrasting colored liquid are in the form of slurries which contain the necessary components for a complete detergent composition.

Referring to the figure, box diagrams **1** and **2** represent the sources of matrix and contrasting colored liquid slurries, **3** and **4**, respectively. These diagrams are intended to include an entire conventional crutching or mixing system together with means for coloring. Conventional crutching systems typically include storage hoppers for raw materials, conveyors, weighing devices, a crutcher, a drop tank, a strainer, a mill and a deaerator.

Where both slurries, **3** and **4**, are complete detergent compositions, they can be prepared in the same crutching system, separated into desired proportions, and one portion given a contrasting color, the colorant usually being added from a reservoir and uniformly mixed with that portion of the slurry before it is sprayed into the tower. As an alternative, it is advantageous to use a matrix which is a complete detergent composition and a contrasting colored liquid which is a slurry of the inorganic components of a detergent composition. In this case, the matrix is prepared in the crutching system and the contrasting colored liquid slurry is prepared in a separate mixing system which is similar to the described crutching system. Generally, crutching can be distinguished from simple mixing in that the crutcher has a jacket through which hot water can be passed in order to maintain the temperature of the mix and an especially effective agitator, which will satisfactorily blend the organic and the inorganic components of the detergent composition.

A typical crutching procedure used to provide the slurry for spray dried detergents is set forth in the examples below.

The uncolored liquid slurry **3** is pumped from the crutching system which is represented by source **1** through conduit **5** by means of booster pump **7** and high pressure (triplex) pump **8** to spray nozzles **9** which are generally present in a plurality of 5 to 12 arranged in the upper portion of spray tower **10** in a circular configuration. Either a ring shaped manifold or separate nozzles can be used. The slurry under pressure is sprayed into tower **10** from spray nozzles **9** in the form of liquid droplets.

The contrasting colored liquid slurry is pumped from source **2** through conduit **6** by means of pump **11**, which is not a high pressure pump. In this case, since lower

pressures are used, a triplex pump can be avoided. The slurry is pumped to nozzle(s) **10** where it is sprayed under pressure into the tower in the form of liquid droplets. Relatively few nozzles, generally one or two, can be used at the lower level, which in this case is about 25% below the level of spray nozzles **9**. The spraying from nozzles **9** and **12** is conducted simultaneously.

The diagram designated air inlet-blower-heater system **13** provides the drying gas, in this case, air, for the countercurrent spray tower system. The warm air is blown into a plenum chamber **14** which is a part of system **13** and through tuyeres **15** into the spray tower upward to meet the droplets from nozzles **9** and **12**, i.e., the air moves in a vertical path countercurrent to the falling droplets, drying the droplets and conventing them to solid particles.

The point of entry of the contrasting colored liquid or the lower nozzle level is defined above. This level is about 15% to 60% below the point of entry of the matrix liquid or upper nozzle level. The percentage is based on the distance from the bottom of the spray tower to the point of entry of the matrix liquid or the upper nozzle level. The upper nozzles are designated as **9** and the lower nozzles as **12**. For the purpose of measuring the distance, the bottom of the tower is defined as the point where the drying gas enters the tower (at tuyeres **15**). The bottom of the tower can also be defined as the lower end of the vertical wall of the spray tower which is in approximately the same position as the air inlet or tuyeres **15** of the countercurrent system. Typical vertical sides measure 60 and 100 feet in length. Preferred percentages for the positioning of the lower nozzle level are about 18% to about 35% lower than the upper nozzle level, with levels of 20% and 33⅓%, being most useful.

The air stream or drying gas carries the smaller fines upward and out of the spray tower to fines system **16** where the reusable portion is returned to the crutching system. The larger fines which take the same path as the particulate product and because of the inventive process do not adhere to the product fall to conveyor **17** along with the product and are separated to some extent in collection system **18**. Fines do appear in final product, however. The various parts of a conventional collection system are vibrating and air conveyors, fan, cyclone, perfume chamber, screens, and receptacle drums not specified in the diagram.

The proportions of matrix and contrasting colored particles in the final product can cover broad ranges and are preferably from about 85% to about 98% by weight matrix particles and about 2% to about 15% contrasting colored particles. Optimum proportions are about 93% to about 97% matrix and about 3% to about 7% contrasting colored particles. The selection of the optimum ranges is usually made by consumer testing to determine the mixture most pleasing to the eye. The proportions are not critical except that the matrix is generally present in an amount at least twice that of the contrasting colored particles in order to achieve the proper effect. The initial materials are selected on the basis of the desired proportions of matrix and contrasting colored particles. Where the same detergent composition except for coloring is used for both, the proportions will approximate the above; where organic and inorganic materials are present in the matrix and only inorganic materials in the colored liquid, there will be some slight variation so that sufficient organic detergent will be present in the final product.

Typical spray tower conditions which can be used to carry out the inventive process are as follows: tower inlet air temperature: 600° to 700° F.; Tower outlet air temperature: 200° to 230° F.; nozzle size (measured by the diameter of the tip orifice in inches): 0.150 to 0.250 in upper level and 0.200 to 0.250 in lower level; number of nozzles: 5 to 12 nozzles at the upper level which are used to introduce the matrix and one or two nozzles at the lower level for introduction of the contrasting colored liquid. The later nozzles(s) can be either introduced into the designated position at the end of a conduit which is called a lance, the lance being inserted from the top of the tower, or a hole can be cut at the desired position in the wall of the tower; upper nozzle pressure 400 to 1000 p.s.i. and preferably 450 to 550 p.s.i., lower nozzle pressure: 60 to 140 p.s.i. and preferabry 90 to 110 p.s.i.

The droplet sizes are important in that fine and relatively coarse droplets sprayed at the different levels provide, as noted, above, a unique feature of this invention. The size of the droplets are determined by the pressure, nozzle orifice size, and nozzle insert and is given in terms of the dried particle since the adjustment of pressure and nozzle is made as a practical matter according to the ultimate particle rather than by measuring the size of the droplet. The upper level pressure and nozzles are adjusted to provide fine droplets and the lower level pressure and nozzle(s) are adjusted to provide relatively coarse droplets, a major proportion of the lower level droplets being larger than a major proportion of upper level droplets. The major proportion and preferably about 60 to 80% by weight, of upper level droplets can be such that in drying particles of −20 to +60 mesh are formed and the major proportion, and preferably about 60 to 80% by weight, of lower level droplets, can be such that particles of −8 to +20 mesh are formed.

Various anionic and nonionic detergents and mixtures of both can be used in the inventive process.

Suitable anionic detergents are water-soluble and have a hydrophobic long chain substituent containing at least 8 carbon atoms, generally 8 to 26 carbon atoms and preferably 12 to 18 carbons, in their molecular structure and at least one water-solubilizing group selected from the group consisting of sulfate, sulfonate, and carboxylate so as to form a water-soluble detergent. The alkyl aryl sulfonates are preferably used as the anionic detergent, the linear alkyl type being preferred over the branched chain. Typical of this class of compounds are those in which the aryl nucleus is derived from benzene, toluene, xylene, phenol, cresol and naphthalene and the alkyl substituents are derived from fatty acids. Examples of the alkyl group are decyl, dodecyl, tridecyl, myristyl and hexadecyl. Mixed long chain alkyls derived from coconut fatty acids and tallow fatty acids can also be used along with cracked paraffin wax olefins and polymers of lower monoolefins. The alkyl groups, as suggested, can be saturated or unsaturated. Examples of other anionic aliphatic detergents are the sulfuric acid ethers of polyhydric alcohols incompletely esterified with higher fatty acids, either saturated or unsaturated, particularly those whose acyl groups contain from 12 to 18 carbon atoms, e.g., coconut oil monoglyceride monosulfate, hydrogenated coconut oil monoglyceride monosulfate, tallow monglyceride monosulfate; the long chain pure or mixed higher alkyl sulfates of 12–18 carbons, e.g., laurylsulfate, cetyl sulfate, higher fatty alcohol sulfates derived from hydrogenated or non-hydrogenated coconut oil or tallow fatty acids; the higher fatty acid esters of hydroxy alkyl sulfonic acids, e.g., higher fatty acid esters of 2,3-dihydroxy propane sulfonic acid, higher fatty acid amides of amino allyl sulfonic acids, e.g., the oleic acid amide of amino methyl sulfonic acid, and the lauric acid amide of taurin. Other aliphatic long chain sul(on)ates can be used including fatty sulfoacetates, e.g., coconut fatty alcohol sulfoacetates; sulfated fatty acyl monoethanolamides, e.g., sulfated lauroyl monoethanolamide; fatty sulfoacetamides, e.g., lauryl sulfoacetamide; lower alkyl sulfosuccinates, e.g., dioctyl sulfo-succinate; sulf(on)ated fatty oils such as sulf(on)ated red oil, and lower alkyl esters of alpha-sulfonated higher fatty acids, e.g., methyl ester of alpha-sulfo myristic acid, sodium salt. Synthetic detergents

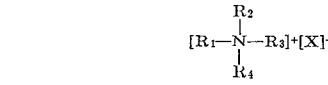
having a carboxylate group and, particularly, the higher fatty acid amides of aliphatic long chain amino acid compounds can also be used such as the higher fatty acyl sarcosinates having about 10 to 18 carbons, usually 12–14 carbons, in the acyl radical, preferably the water-soluble salts of N-lauroyl or N-cocoyl sarcosine. Other materials are higher fatty acid amides of polypeptide amino acids obtained by protein hydrolysis. Suitable ether-containing sulfates which can be used are the alkylphenol polyglycol ether sulfates, e.g., lauryl phenol polyethyleneoxy sulfates and alkyl polyglycol ether sulfates, e.g., lauryl ethyleneoxy sulfates, each containing about 18 to 18 carbons in said alkyl groups and about 2 to 10 moles of ethylene oxide, usually 3–4 moles, per molecule. These various anionic detergents are used in the form of their water soluble or water dispersible salts such as the amine, alkali metal and alkaline earth metal salts. Examples are the sodium, potassium magnesium salts, ammonium, monoethanolamine, diethanolamine, triethanolamine salts, and mixtures thereof.

As the nonionic detergent, any of the conventional water-soluble nonionic detergents can be used in the composition of this invention. These nonionics can be in liquid or paste form.

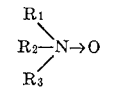
Generally, such nonionics have a hydrophobic group containing at least 8 carbon atoms and preferably 8 to 30 carbon atoms. One particular class of such detergents is that formed by the oxyalkylation of fatty acids, alcohols, phenols, mercaptans, thiophenols, amines and amides with ethylene oxide, propylene oxide, and other related alkylene oxides. Such materials usually have at least 5 mols of alkylene oxide, and preferably 5 to 30 mols of alkylene oxide, depending upon the particular hydrophilic group desired. Representative of these materials are those formed by the condensation of ethylene oxide with alkyl phenols or alcohols. Particularly preferred herein are condensates formed by the reaction of one mole of nonyl phenol or a mixture of C_{12}–C_{18} saturated, straight-chain, aliphatic alcohols with 8 to 12 mols of ethylene oxide, the condensates containing an average of about 8 to 10 ethylene oxide groups per molecule. Some specific examples of this type of nonionic detergent are as follows: nonyl phenol-ethylene oxide condensates having an average of 9.5 ethylene oxide groups per molecule; a mixture of saturated aliphatic alcohols having from 14 carbons to 18 carbons in their chains and an average of 8.5 mols of ethylene oxide per molecule; tallow alcohol-ethylene oxide condensate having an average of 9 mols of ethylene oxide per molecule; and a 1:1 mixture of C_{12} and a C_{14} saturated aliphatic alcohol having an average of 8.5 mols of ethylene oxide per molecule. Other alkylphenol condensates are those of diamylphenol, p-tert-octylphenol, 2,4 - dicyclohexylphenol, m - pentadecylphenol, and benzyl-o-hydroxybiphenyl. Other condensates with alkylene oxide are those of tall oil, branched chain C_{14} to C_{17} aliphatic alcohols, lanolin, beeswax, bisphenols, oxidized paraffin wax, naphthenic acids, and fatty acyl alkanolamides. Mixtures of various water soluble nonionic detergents are contemplated.

The builder salts which can be used in this process are well known materials. Both inorganic and organic, basic and neutral water soluble salts are included in this group. Among the inorganic builder salts, the phosphate builders are in common usage. Examples of phosphate builders are the alkali metal tripolyphosphates and pyrophosphates of which the sodium and potassium salts are generally used either alone or in admixture. Other examples of phosphate builders are sodium tripolyphosphate; sodium phosphate, tribasic; sodium phosphate, monobasic; sodium phosphate, dibasic; sodium pyrophosphate; and sodium pyrophospate, acid. The corresponding potassium salts can also be used along with mixtures of the salts or corresponding mixed potassium-sodium salts. Of the tripolyphosphates, a low phase 1 material is preferred and is conventional for spray drying operations. The low phase 1 material is a phase 2 tripolyphosphate associated with a maximum of about 8% phase 1 tripolyphosphate, the phase 1 crystalline form being a high-temperature-rise material which hydrates more rapidly than the phase 2 material. Other inorganic builder salts are exemplified by alkali metal sulfates such as sodium sulfate; sodium chloride; sodium carbonate; sodium metasilicate; sodium silicates wherein the ratio of Na_2O to SiO_2 is from about 1:1.6 to about 1:3.2; borax; and ammonium carbonate. The potassium ion can be substituted for the sodium ion wherever feasible and mixtures of the foregoing builder salts are contemplated. The organic builders include salts of organic acids and, in particular, the water soluble salts of aminopolycarboxylic acids. The alkali metal salts such as sodium, potassium, and lithium; ammonium and substituted ammonium salts such as methyl ammonium, diethanolamonium, and triethanolammonium; and amine salts such as mono, di, and triethanolamine, methylamine, octylamine, diethylenetriamine, triethylenetetramine, and ethylenediamine are efficacious. The acid portion of the salt can be derived from acids such as nitrilodiacetic acid; N-(2-hydroxyethyl) nitrilodiacetic acid; nitrilotriacetic acid (NTA); ethylenediaminetetracetic acid (EDTA); N-(2-hydroxyethyl) ethylene diamine triacetic acid; 2-hydroxyethyl iminodiacetic acid; 1,2-diaminocyclohexanediacetic acid; diethylenetriamine penta-acetic acid.

Softeners such as quaternary ammonium salts can be included in the process. They are exemplified by the following general formula:



wherein R_1 and R_2 are alkyl groups each containing from 1 to 3 carbon atoms; R_3 and R_4 are aliphatic groups each containing from 12 to 22 carbon atoms; and X is selected from the group consisting of chlorine, bromine, and methyl sulfate. These compounds are readily dispersible in water. Specific examples are as follows: distearyl dimethyl quaternary ammonium chloride; distearyl dimethyl quaternary ammonium bromide; distearyl dimethyl quaternary ammonium methylsulfate; dicoco dimethyl quaternary ammonium chloride; dimethyl arachidyl behenyl quaternary ammonium chloride; dialkyl dimethyl quaternary ammonium chloride, the alkyl groups of which comprise a mixture consisting essentially of 24 parts hexadecyl, 75 parts octadecyl, and 1 part octadecenyl groups; the latter quaternary ammonium chloride is also known as dimethyl dihydrogenated ditallow ammonium chloride and is particularly preferred. Mixtures of two or more cationic softener agents can be employed if desired. (The term "coco" refers to fatty acid groups formed in coconut oil fatty acids. Such acids contain from about 8 to about 18 carbon atoms per molecule, predominating in C_{12}–C_{14} acids.) Other examples are dimethyl ditallow hydrazinium chloride and dimethyl ditallow quaternary ammonium methyl sulfate. It should be noted that the quaternary salts can only be used with nonionics in softener-detergent compositions. Other softeners which can be used with anionic and nonionic detergents are exemplified by the amine oxides characterized as mono, higher alkyl, di-lower-alkyl or hydroxy-alkyl amine oxides of the general formula:



wherein R_1 is an alkyl of at least 18 carbon atoms and preferably 18 to about 22 carbon atoms and R_2 and R_3 may be independently lower alkyl or hydroxyalkyl of from 1 to 4 carbon atoms, and a water-soluble builder.

Among the amine oxides contemplated are:

stearyl dimethylamine oxide
stearyl diethylamine oxide

stearyl di-n-propylamine oxide
stearyl di-isopropylamine oxide
stearyl di-n-butylamine oxide
stearyl di-sec-butylamine oxide
stearyl di-tert-butylamine oxide
eicosyl-1-dimethylamine oxide
docosyl-1-dimethylamine oxide
stearyl, methyl, ethyl amine oxide
stearyl, methyl isopropyl amine oxide
stearyl ethyl, isopropyl amine oxide
stearyl isopropyl, isobutyl amine oxide
docosyl methyl ethyl amine oxide
docosyl diethylamine oxide
docosyl diisopropyl amine oxide
stearyl bis(2-hydroxyethyl)amine oxide
stearyl methyl-2-hydroxyethyl amino oxide

While those amine oxides which are preferred are as illustrated above, one can also employ as the R_1 radical a mono- or di-unsaturated moiety provided, however, that for each degree of unsaturation there is present one extra carbon atom above the critical minimum of 18 carbon atoms. Thus R_1 may also be eicosenyl, docosenyl, or tricosenyl.

Various other additives can also be used in the inventive process. Alkali metal carboxymethylcellulose can advantageously be introduced as well as perfumes, brighteners, and bluing agents. Examples of brighteners are stilbenes, e.g. triazoles and cyanurics, and benzidine sulfones, the stilbene triazoles being preferred. An example of a bluing agent is ultramarine blue, which can be used to serve a dual purpose, i.e. colorant and bluing agent. A total laundry care composition can also be prepared where it is desired to include bleaching agents with the aforementioned components. Generally the bleaching agents are oxygen bleaches typified by sodium and potassium perborates and potassium persulfate. Soil suspending agents such as polyvinyl alcohol as well as antioxidants can also be included if desired.

The proportions of components which are typically present in the final product, in parts by weight, are as follows: detergent—about 5 to about 30 parts and preferably about 7 to about 15 parts; quaternary ammonium salt—about 2 to about 8 parts and preferably about 3 to about 6 parts (where a softener-detergent composition is desired); builder salt—about 15 to about 87.5 parts and preferably about 60 to about 85 parts; alkali metal carboxymethylcellulose—about 0.1 to about 5 parts and preferably about 0.2 to about 3 parts; colorants, perfumes, brighteners, bluing agents, soil suspending agents, soaps and antioxidants—total weight 0 to about 4 parts and preferably 0 to about 3 parts; bleaching agent—0 to about 10 parts, preferably 0 to about 5 parts (where total laundry care is desired).

There is usually about 1 to about 15% by weight water and preferably 7 to 12%. The preferred water contents depends on the material to be spray dried, organic materials being dried to a 1% or less water content. However, substantial drying is considered to be about 15% or less.

Heat stable colorants must be selected for use in the within process since the coloring materials are exposed to the heat of spray drying. The colorants are desirably alkaline stable and have negligible or no substantivity to fabrics. They can be dyes, inorganic pigments or organic pigment. The following is a list of some suitable coloring agents including both dyes and pigments:

Blue ——— Phthalocyanine Blue (Monastral Fast Blue, Heliogen Blue), Ultramarine, Patent Blue A, Erioglaucine, Indigo, Brilliant Blue FCF.
Red ——— Madder Lake, Rhodamine Pigment, Crimson EMB, Lithol Rubine, Azo Rubine.
Orange ——— Chrome Orange, Alizarin.
Yellow ——— Indanthrene Yellow, Chrome Yellow, Naphthol Yellow, Metanil Yellow, Tartrazine, Hansa Yellow.
Green ——— Phthaocyanine Green (Monastral Fast Green, Heliogen Green), Pigment Green B, Naphthol Green B, Alizarin Cyanine Green.
Violet ——— Wool Violet, Anthraquinone Violet.

The coloring materials can be used as solutions or suspensions in a suitable solvent, usually water. However, any solvent can be used in suitable concentration which would not present an explosion hazard in the operation of a spray-tower.

Usually, the solutions or suspensions of coloring material contain about one to about five percent by weight of coloring material.

The following examples in which parts and percentages are by weight are illustrative of the invention.

EXAMPLE I

This example is carried out using the apparatus described above.

The crutcher mix is as follows:

Component:	Parts
Water	38.0
Linear tridecyl benzene sulfonate	10.0
Ethoxylated C_{14}–C_{15} linear alcohol containing 11 ethylene oxide groups	2.0
Sodium soap	2.0
Sodium silicate	7.0
Sodium carboxymethyl cellulose	0.5
Polyvinyl alcohol	0.2
Sodium tripolyphosphate	34.0
Brighteners, antioxidant, etc.	0.45
Ultramarine blue	0.15
Sodium sulfate	35.0

Steam at atmospheric pressure is directed continuously against the jacket of a crutcher which is equipped with a paddle mixer. Water is introduced into the crutcher at about 120° F. The detergent is then added without agitation and medium agitation of 150 r.p.m. is then commenced.

Then the ultramarine blue, sodium carboxymethylcellulose, brighteners, polyvinyl alcohol, and antioxidant are added. After which, sodium sulfate and sodium silicate are added. The temperature is then raised to 150° F. followed by the introduction of the sodium tripolyphosphate with an increase in agitation to about 350 r.p.m. Crutching time is about 15 minutes. The resulting slurry is then separated into 95 parts matrix (light blue) and 5 parts for coloring. The 5 parts is then mixed with .075 part phthalocyanine blue to obtain a dark blue color.

The light blue slurry is then pumped to the upper nozzle level and the dark blue slurry to the lower nozzle level under the following spray tower conditions:

Tower inlet air temperature—about 650° F.
Tower outlet air temperature—about 215° F.
Nozzle tip orifice—upper level—.177 inch
Nozzle tip orifice—lower level—.213 inch
Number of nozzles—upper level—6
Number of nozzles—lower level—1
Pressure—upper level—700 p.s.i.
Pressure—lower level—100 p.s.i.
Particle size distribution in final product resulting from upper level:

U.S. Sieve No.:	Percent
+8	1.5
−8 +20	27.0
−20 +40	42.0
−40 +60	20.0
−60 +80	4.0
−80 +100	2.0
−100	3.5

Particle size distribution in final product resulting from lower level:

−8 +20	greater than 60 percent
−20 +40	greater than 20 percent

Note.—The designation of minus means material passing through the mesh screen and the designation of plus means material caught on the mesh size screen.

	Feet
Distance from upper level to bottom of tower	60
Distance from upper level to lower level	20

The resulting product is a homogeneous mixture of 95 parts light blue matrix particles and 5 parts dark blue particles which are substantially distinctive against the background matrix. The particles have a moisture content of 8.5% and are discrete with substantially no fines adhering to the surface of the dark blue particles and no agglomeration.

EXAMPLE II

Example I is repeated except that a slurry of inorganic components only is mixed separately, colored, and sprayed from the lower nozzle. The light blue slurry is used in an amount of 96 parts and the dark blue inorganic slurry is used in an amount of 4 parts. The components of the dark blue slurry are silicate, sulfate, tripolyphosphate, and phthalocyanine blue in the same proportions as in Example I.

The result is the same as described in Example I except for the slight difference of proportions between the light blue (96%) and dark blue (4%) particles.

EXAMPLE III

Example I is repeated except that ultramarine blue is added to the dark blue slurry. The matrix is, therefore, white.

The result is the same as in Example I except that the resulting matrix particles are white.

EXAMPLES IV and V

Example I is repeated omitting in one case the sulfonate detergent and in the other case the ethoxylated alcohol detergent maintaining however the same total amount, i.e., 12 parts of detergent.

The results are the same as in Example I.

EXAMPLE VI

Example III is repeated except that the phthalocyanine blue is omitted from the dark blue slurry. The only colorant is ultramaine blue and, therefore, the shade of blue is not as dark.

The result is the same as in Example III except for the shade of blue of the colored particles.

EXAMPLE VII

Example I is repeated except that no dyestuff is added to either the 95 part sample of matrix or 5 part sample of matrix. The results obtained are similar to those described in Example I i.e., the particulate product is substantially free from fines and agglomeration.

What is claimed is:

1. A process for the continuous production of a particulate product in a countercurrent spray drying system comprising the following steps:

(a) forming a first stream of liquid matrix capable of being converted into solid particles by spray drying;

(b) passing the first stream to a predetermined point of entry in the upper portion of a spray tower;

(c) forming a second stream of a liquid matrix and capable of being converted into solid particles by spray drying;

(d) passing the second stream to a point of entry in the spray tower at a level below that of the point of entry of the first stream at which substantially all of the liquid particles formed at the point of entry of said first stream have been substantially dried, and, at a level above the bottom of the spray tower sufficient to substantially dry droplets formed by the second stream;

(e) passing a stream of drying gas upwardly through the tower;

(f) simultaneously spraying the two streams from their respective points of entry downwardly in the form of a plurality of liquid droplets into the upwardly flowing stream of drying gas, whereby the droplets are dried and converted to solid particles; and

(g) removing the dried product containing a homogenous mixture of substantially discrete particles from the drying gas.

2. The process defined in claim 1 wherein the point of entry of the second stream is about 15% to about 60% below the point of entry of the first stream, the percentage being based on the distance from the bottom of the spray tower to the point of entry of the first stream.

3. The process defined in claim 2 wherein the droplets resulting from the first stream are relatively fine droplets compared to the droplets resulting from the second stream.

4. The process defined in claim 3 wherein the fine droplets are of a size sufficient to provide a major proportion of particles having a size distribution between about −20 and 60 mesh and the droplets produced from the second stream are of a size sufficient to provide a major proportion of particles having a size distribution between about −8 and 20 mesh.

5. The process defined in claim 2 wherein the liquids are detergent compositions containing a member selected from the group consisting of anionic and nonionic detergents and mixtures thereof.

6. The process defined in claim 5 wherein the detergent composition additionally contains a builder salt selected from the group consisting of organic and inorganic builder salts.

7. The process defined in claim 2 wherein the second stream has an inorganic composition comprising inorganic builder salts.

8. The process defined in claim 2 wherein said first and second streams are of contrasting color.

References Cited

UNITED STATES PATENTS

2,232,544	2/1941	Lovenz	240—418
2,579,944	12/1951	Marshall	159—48 X
2,701,262	2/1955	Cook	260—555
2,842,193	7/1958	Ballestra	159—4
3,121,639	2/1964	Bauer et al.	99—203
3,143,428	8/1964	Reimels et al.	159—4 X
3,296,240	1/1967	Mac Donald et al.	260—93.7
3,357,476	12/1967	Tofflemire	159—4

NORMAN YUDKOFF, Primary Examiner

J. SOFER, Assistant Examiner

U.S. Cl. X.R.

159—4; 252—110, 137