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[54] **MULTILEVEL SPRAY-DRYING METHOD**
10 Claims, 2 Drawing Figs.

[52] **U.S. Cl.** ..... **34/33,** 34/168, 34/174, 159/4 CC, 159/DIG. 14
[51] **Int. Cl.** **F26b 3/00**
[50] **Field of Search** ..... 34/31, 33, 168, 174; 159/4 CC, DIG. 14

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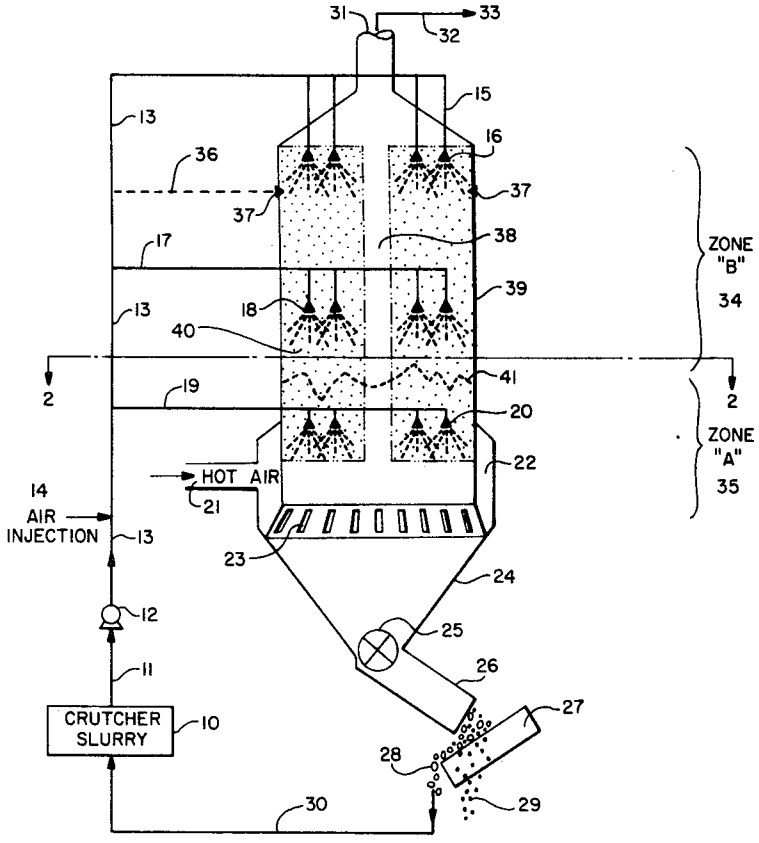
ABSTRACT: A method is provided for spray-drying large volumes of a synthetic detergent slurry which comprises spraying the slurry into the spray-drying chamber in at least two different levels of uniformly spaced atomizing nozzles. The lowest level of nozzles is critically positioned at a point in the spray chamber below a 190° F. isotherm and above a boiling point isotherm. From 30 to 80 percent of the slurry is atomized at this lowest level. The balance of the same slurry is sprayed through the remaining levels. Apparatus is provided for practicing this process.



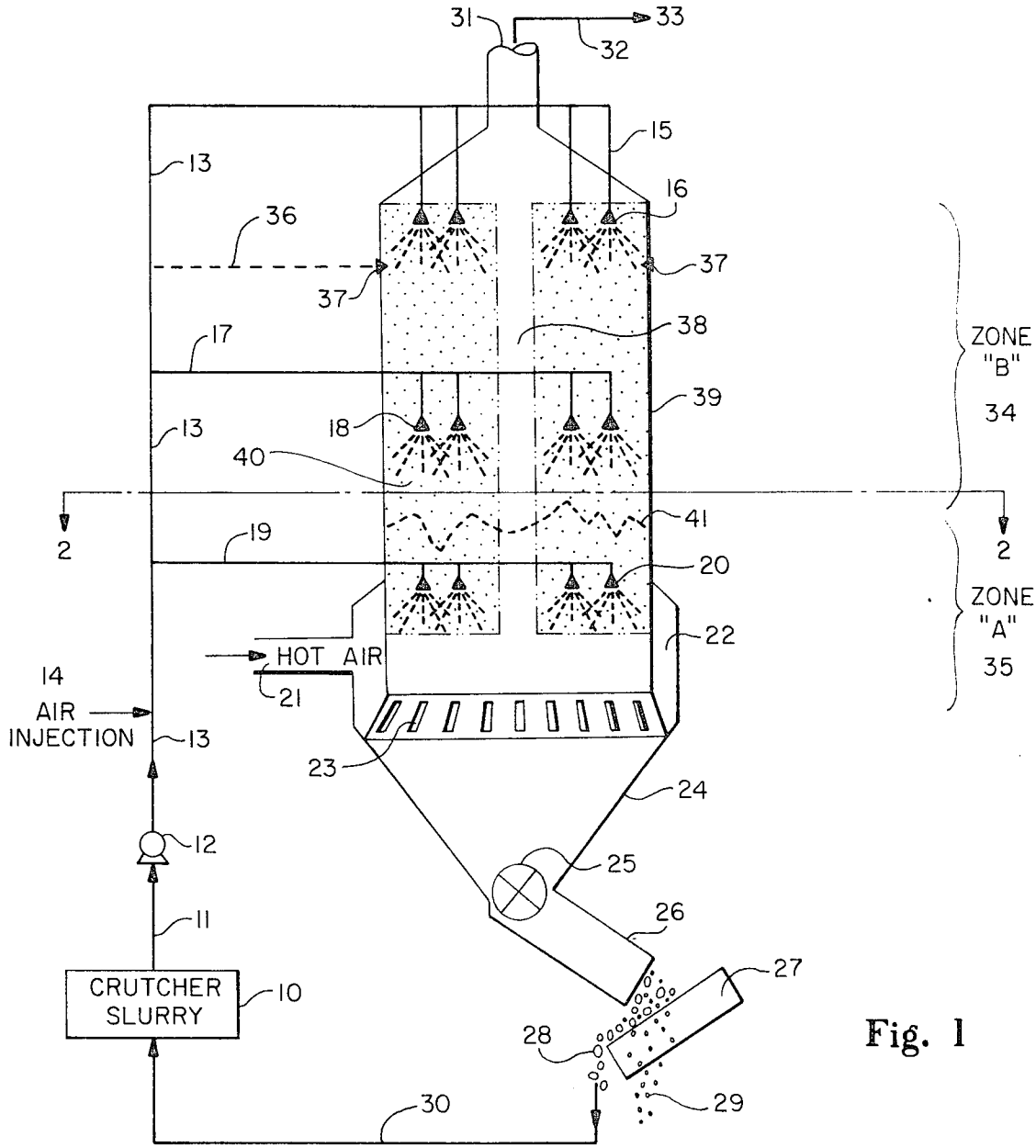


Fig. 1

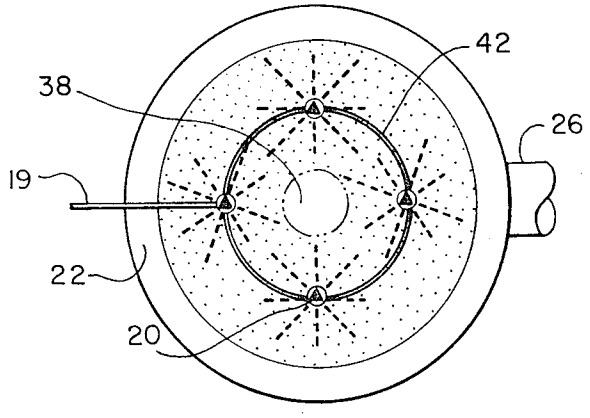


Fig. 2

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MULTILEVEL SPRAY-DRYING METHOD

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to the art of spray-drying synthetic detergent aqueous slurries to form granular synthetic detergent compositions.

2. Description of the Prior Art Practices

This invention relates to a large volume operation involving spraying thousands of pounds per hour, hundreds of thousands of pounds per day. Ordinarily spray towers are used employing a single level of atomizing nozzles which are located near the top of the spray-drying chamber. Spray-drying large volumes of detergent slurries is a complex procedure involving numerous interrelated factors such as volume and rate of production, different ingredients which comprise a synthetic detergent slurry, different processing requirements and conditions, different characteristics of the numerous ingredients, e.g., hydration properties, massive requirements of drying air, the desired physical and performance properties of the eventual spray-dried product, leveling and packaging of final spray-dried granular product.

SUMMARY OF THE INVENTION

It has now been discovered that an ordinary single-level spray-drying method can be substantially improved with surprising and unexpected results by employing multilevels of atomizing nozzles provided that certain important horizontal and vertical alignments are met together with compliance of important processing conditions.

As a result of practicing this invention, it is possible to significantly increase the rate of production over ordinary single-level spray-drying operations. Improved rates can, for example, be on the order of 10–30 percent. In the context of large volume productions such magnitudes of rate improvement can represent millions of pounds annually.

Surprisingly this increase in production rate is achieved without resort to more severe heat requirements. It was completely unexpected that the present multilevel spray-drying process would provide such increased efficiency in heat utilization.

The present method has an another objective the advantage of providing a significant measure of control over the density of the final dried granules. By adhering to the teachings of this invention, it is possible to decrease the density of certain compositions and to increase the density of other products. While the most frequent objective in ordinary commercial practices is to produce granules of decreased density, the present invention provides a reasonable degree of flexibility in achieving greater densities also.

Surprisingly, the decrease in particle density is achieved even though the average particle size is uniformly smaller. Normally, the finer the spray-dried particle size, the greater the density. Inspection of product sieve fractions from the practice of this invention indicates that the specific density of the individual particles are significantly lower and that the individual granule shape is irregular. It is speculated that the combination of these two observations offset the normally expected density increase normally associated with overall finer granular product.

Another unexpected advantage of the present invention is the substantial decrease in the amount of fine powders and vaporous effluent materials produced by the method of the present invention. Reductions as high as 50 percent have been found. The advantage of such an improvement in terms of environmental control, i.e., air pollution is noteworthy. Not only is fine powder production cut in half at the exhaust tower, but there is additional improvement in the marked decrease in aerosol (vapor) contaminants which pass into the atmosphere. This improvement is one which is more difficult to measure and lends itself more readily to subjective appraisal. Nevertheless, the improvement is real and can represent a significant advance in efforts to improve and comply with clean air standards. The aerosol and vaporous effluents have the tendency to give permanence to steam and smoke plumes occasionally seen coming from spray tower facilities.

Moreover, in addition to the marked reduction of fine powders (overhead dusts) the present invention provides an equally significant reduction in heavy coarse products (tower tailings). Consequently, by minimizing production of fine powders and coarse granules, the manufacturer is able to enjoy a proportional improvement in product satisfactory for packing. This provides an ultimate economic savings of substantial magnitude in the context of the huge amounts of production contemplated.

A further unexpected result of the present invention is that each of the aforementioned advantages are provided without increasing the amount of insolubles formed by the spraying operation. Such insolubles are at times referred to as floc and are formed it is believed by physical and chemical degradations due to severe drying conditions. An essential embodiment of the present invention as described below comprises spraying a very substantial proportion of the detergent slurry into a high-temperature zone that was heretofore intentionally avoided by widely practiced commercial spray-drying procedures. Thus it was expected that the exposure of freshly sprayed droplets to inordinately high temperatures would cause excessive formations of floc and insolubles. This does not occur, however.

While the role of phosphates in detergent compositions is being questioned in terms of water quality, the outcome is still in doubt. In any event the present invention provides a significant advance in spray-drying phosphate builders such as sodium tripolyphosphate. For a long time, one of the widely held serious limitations in using higher spray-drying temperatures for phosphate-containing detergent compositions was that overdrying caused a marked reversion of phosphates to other less desirable phosphorus compounds such as pyrophosphates and orthophosphates. These latter materials are admittedly poorer detergency builders. The present multilevel spray-drying method is not handicapped by problems of such reversion. In fact, there is less reversion with the present invention than one finds with an ordinary single-level spray-drying process.

Several unexpected advantages enjoyed by the present method are attributed to the overall less severe drying conditions which are employed by the present invention. In this respect, one of the major concerns in an ordinary single-level spraying operation is overdrying the freshly sprayed particles as they dry falling through the tower. Ordinarily the hottest zone, the area where the highest isotherms exist, is near the lowest region of the spray chamber. This is the point at which hot air is introduced and dispersed through plenum arrangements. The heated drying gas passes up through the tower countercurrently to the falling atomized particles. As the atomized droplets fall through the rising air currents, they begin to dry. However, the removal of water is relatively slower in the upper tower regions which, while warm, is still cooler than the hotter lower regions. By the time the droplets fall into the highest temperature zone, they have dried sufficiently to have set and solidified to form granules having a hard surface skin. It is these dried particles which by ordinary conventional practice must still pass through the highest temperature zone. It is here that overdrying problems can occur. Phosphate reversion is just one of these problems. The degradation of other detergent additives such as brighteners, amides, nonionic detergents, germicides is also known to occur in this region. Such degrading action not only can adversely affect the overall performance effectiveness of the products but also give rise to unpleasant color and odor problems and other aesthetic negatives.

Surprisingly these overdrying problems are considerably alleviated by the present invention. The exact reason for this is not known. What is believed to occur however, and this is reasoned speculation, is that atomization of a portion of the crutcher mix into a zone intermediate a 190° F. isotherm and a boiling point isotherm (the remainder of the mix being

atomized into zones of still lower temperature) results in a less severe timeltemperature exposure for the resulting product. In addition, there is a sudden release of steam and gases, not heretofore experienced in prior art spray-drying operations, which tend to alter and beneficially affect rising air currents. Thus, the particles freshly sprayed into the top of the spray tower fall through and are exposed to a drying environment materially different from anything previously known. The consequences are all of the beneficial results noted above as well as the very significant improvement described below respecting spray-drying synthetic detergent compositions containing low levels of phosphate builders or detergent compositions in which the phosphate builder has been replaced partially or completely with a phosphate-free builder such as sodium nitrilotriacetate (NTA), sodium citrate, sodium mellitate, sodium oxydiacetate, starch, cellulose, sugars and sugar derivatives, sodium oxydisuccinate and the like.

Because of the predominant role which sodium tripolyphosphate has held as a detergency builder over the last three decades, the bulk of spray-drying technology has developed naturally around that single builder material. Now that considerable emphasis is being placed on finding partial or total replacement of phosphates, the known spray-drying techniques are being applied to new materials.

In arriving at the present invention, it has been discovered that phosphate spray-drying technology does not uniformly apply to sodium nitrilotriacetate and other phosphorus-free builder systems (the term systems meaning broadly other sole builder replacements or mixtures of such alternative materials). It was disappointing to discover that existing factory facilities and supporting engineering resources could not carte blanche be applied to, for instance, NTA-built systems.

One of the more important objectives of this invention therefore is to provide a method and apparatus that successfully solves the several problems encountered in spray-drying nitrilotriacetate builders, as sole detergent ingredients or as mixtures with sodium tripolyphosphate. One severe obstacle encountered in spray-drying NTA/STP blends was the stickiness of the resulting granules. Handling such granules presented a large problem. Transporting them, storing them and packaging them was difficult. The present invention, methodwise and apparatuswise, solved each of these problems in a highly reasonable and satisfactory manner.

As a a result, whereas it was thought that major production rate cutbacks would be necessary with novel-built detergent compositions (i.e., other than phosphate builders), the present invention provides high-volume production of crisp, controlled density, uniformly sized granular synthetic detergent compositions.

The foregoing objects and improvements are achieved by the present invention which in its method embodiments comprises a continuous method for spray-drying large volumes of a synthetic detergent slurry in a spray-drying tower and producing a granular synthetic detergent composition having controlled density and uniform reduced particle size with minimum production of dust particles and other vaporous effluents comprising the following steps:

1. preparing an aqueous synthetic detergent slurry having about 10–50 percent by weight water and the balance 50–90 percent solids content being comprised of at least one organic synthetic detergent, and at least one detergency builder selected from organic or inorganic builders or mixtures thereof;

2. establishing within the chamber of the spray tower (a) a cylindrically shaped drying zone with the axis of the chamber by passing heated drying air upwards through the chamber in a cyclonic motion and (b) a low-pressure zone comprising a concentric vortex tube which is formed along the axis of the chamber;

3. continuously spraying countercurrently from 30 to 80 percent of said synthetic detergent slurry directly into the cylindrically shaped drying zone at the point below a 190° F. isotherm and above a boiling point isotherm said spraying being achieved with atomizing nozzles substantially uniformly spaced in a horizontal plane through the cylindrical drying zone thereby providing that substantially each of the sprays disintegrates into particles within said cylindrical drying zone;

4. continuously spraying countercurrently the balance of said synthetic detergent slurry directly into the cylindrically shaped drying zone at a point above the 190° F. isotherm by means of at least one level of atomizing nozzles substantially uniformly spaced in a horizontal plane through the cylindrical drying zone, thereby providing that substantially each of the sprays disintegrates into particles within said cylindrical drying zone, whereby the only disintegrated particles entering the low-pressure vortex tube are those incidentally carried by the cyclonic motion of the drying gas.

The apparatus aspects are apparent from the detailed discussion below:

DRAWINGS

Attention is drawn to the two figures comprising part of this application.

FIG. 1 is a side elevational view illustrating a multilevel spray-drying tower incorporating the present invention.

FIG. 2 is an enlarged cross-sectional detail taken along line 2—2 of FIG. 1 and serving to illustrate the cylindrically shaped drying zone, the concentric vortex tube, and the manner in which the atomizing nozzles are substantially uniformly spaced in a horizontal plane through the cylindrical drying zone.

The spray-drying tower apparatus illustrated in the drawing is now described in order to present both the apparatus embodiments and method embodiments of the present invention.

Referring to FIG. 1, box diagram **10** represents a crutcher slurry preparation. This is intended to include an entire conventional crutching or mixing system together with means, **11**, for passing it to a high-pressure pump, **12**. Conventional crutching systems are well familiar to those skilled in the art and typically include storage hoppers for raw materials, conveyors, scales, a crutcher, a drop tank, and the like. For purposes of the present invention, the slurry is comprised of 10 to 50 percent water by weight and the balance 50 to 90 percent solids content. The solids content is made up of the ingredients which constitute the formula for the desired granular synthetic detergent composition. The crutcher slurry contains at least one organic synthetic detergent of an anionic, nonionic, ampholytic, or zwitterionic type; preferably anionic detergents are employed. A detailed description of suitable detergent materials is given hereinafter. At least one detergency builder is added to the crutcher slurry. It can be of an organic or inorganic type, again as described in detail elsewhere in this specification. It is common to employ mixtures of different detergents and different builder materials in preparing the slurry.

The slurry is passed through suitable pipes, conduits and the like designated at **11** by means of a high-pressure pump, **12**. Any suitable pump can be used by preferably those capable of providing pressure in the range of 400 to 2,000 p.s.i.

Although the invention is susceptible of variation and adaptation with respect to many of the particulars such as the flow ducts, an air injection system is shown at **14**. Basically this is a traditional density control means rather universally employed. While this is an optional embodiment in terms of this invention, it is a helpful device and its employment is recommended. The amount of air injection into the system from this ancillary source should range from 0 to 150 standard cubic ft./min., and preferably 0 to 100 standard cubic ft./min.

From the air injection step, the aerated slurry is passed to the spray-drying tower chamber, **39**, simultaneously by feedline **13** to nozzle arms **15** and atomizing nozzles **16**, by feedline **17** to atomizing nozzles **18**, and by feed **19** to atomizing nozzles **20**.

The spray-drying tower is illustrated as comprising a spray-drying chamber **39**, having the atomizing nozzles uniformly

and discretely spaced therein; a hot air duct **21**, passing to a plenum **22** for distributing the hot air into the chamber **39** by a means of tuyeres **23**. The hot air by this arrangement, and this is critical to the optimum practice of the present invention, is introduced into the chamber **39**, in the form of cyclonic motion. For best results the hot air should have a temperature in the range of 300° to 800° F., preferably 400° to 700° F. and be introduced at a rate of 1,000 lb./min. to 6,000 lb./min. preferably 2,000 to 4,000 lb./min. The cyclonic motion of the heated drying air has an important bearing on the vertical spacing of the multilevels of nozzles **16**, **18**, and **20**, as well as the horizontal spacing of the atomizing nozzles uniformly within each spraying level.

At the base of the spray tower is a cone **24**, valve **25**, and conveyor means **26**, by which the dried granules are removed. The conveyor means **26**, passes the dried granules to a sifting screen **27**, at which point coarse granules **28**, are gathered and can be recycled by line **30** to the crutcher slurry, **10**. The desired product granules **29** are collected and packaged or stored.

The top of the spray tower is equipped with exhaust means **31**. Leading from the exhaust exit is a line **32** designated to lead fine particles to an appropriate treatment or recovery area **33**. From this point the spent exhaust gases are passed into the atmosphere.

Within spray chamber **39** there is designated a cylindrical spray-drying zone **40** and a vortex tube **38**. The parameters for the cylindrical spray-drying zone **40** and the vortex tube **38** are determined by the cyclonic effect of the rising heated air. It is important to the practice of this invention that the sheets of sprays from the atomizing nozzles disintegrate in the designated cylindrical drying zone. It has been discovered that if this condition is met, the optimum results are obtained in terms of increased production rates, controlled density, uniform particle size, reduced stickiness of the granules, reduced production of fine (dust) and coarse granules, and reduced vaporous effluents. The size of the vortex tube can vary depending on several factors including velocity of the cyclonic heated drying air, size of apparatus etc. The important consideration with respect to the vortex tube is that it is an area of decreased pressure and any particles freshly sprayed into this vortex tube area are not subjected to the desirable optimum drying influences created by the critical horizontal and vertical alignment of the levels of nozzles as well as their critical uniform horizontal spacing within each level.

Freshly sprayed particles entering into the low-pressure region of the internal concentric vortex tube fall prematurely through the tower and interfere with the objectives of the inventions identified above. It was consequently discovered that, in addition to a critical vertical spacing of the levels of atomizing nozzles discussed below, the horizontal spacing of the nozzles must be such that the sheets of spray from each nozzle must disintegrate within the prescribed cylindrical spray-drying zone; care must be taken that the sheets of spray are not sprayed into the vortex tube. It is in this context that the term "directly into the cylindrical spray-drying zone" is used to indicate the importance of avoiding spraying into the vortex tube area.

FIG. **1** also embodies another essential embodiment of this invention, namely the vertical spacing of the plurality of levels of spray nozzles. Special consideration is to be given the lowest level of the spray nozzles for its positioning is fundamental to achieving and optimizing the objectives noted above. In FIG. **1**, the lowest level is designated by feedline **19** and atomizing nozzles **20**.

This lowest level of spray nozzles is essentially located at or below a 190° F. isotherm, **41** and above a boiling point isotherm. Isotherms are well understood temperature profiles within a spray-drying chamber involving heated drying air. It is necessary that the freshly sprayed particles at this lowest level be exposed to temperatures in the range of 190° F. to about 210°–220° F. This permits rapid puffing of the granules with a corresponding reduction in density. The particle size is controlled because the rapid evaporation which occurs is not so rapid as to explode the granules and produce inordinate amounts of fine powders. Large amounts of fines would tend to increase the density of the final granular product. In addition to the release of substantial amounts of water in this space as a result of rapid drying, there is also a significant production of expanding and released gases. Both the released water in the form of steam and the released gases pass up through the tower with the heated drying gas. This type of a dynamic system has not previously been known. The beneficial effects have never previously been recognized.

Referring to FIG. **1** it is seen that the 190° F. isotherm, **41** and the lowest level of spray nozzles are positioned in zone "A" **35**. The size of this zone is, of course, susceptible of variation and modification due to adjustment of any of several processing variables. The significance of designating the 190° F. isotherm, **41** and the lowest level of spray nozzles **20**, is to emphasis the essential space relationships of these two factors. The balance of the spray tower is designated as zone B, **34**. In this region the drawing illustrates two levels of spray nozzles, **16** and **18**. It is to be noted that while two levels are shown, only one needs to be present to provide the benefits of this invention. Thus it is within the contemplation of this invention to embody as few as two levels of spray nozzles, for example, **16** and **20**, or **18** and **20**. It is possible, however, to have levels of nozzles in zone B, **34** spaced at 8-foot intervals. Thus, if zone B were 50 feet high there would be space for as many as six levels of atomizing nozzles. In any event, an essential feature is to provide means for spraying from 30–80 percent of the detergent slurry in zone "A" **35**, i.e., below a 190° F. isotherm and above a boiling point isotherm. The reason for remaining above a boiling point isotherm has been implied above. Exceeding the boiling point of the slurry would have an adverse effect on the drying rate, production of fines and possible charring of the product.

It is necessary to provide at least 30 percent of the slurry into the lowest level to obtain the maximum benefit of the invention. While amounts greater than 80 percent can be fed to this level, it is preferred to remain below 80 percent to balance the several processing conditions involved, rate of addition of the heated drying gas, the cyclonic effect, the rate of drying and the like. Optimum results are obtained when from 35–70 percent by weight is sprayed into the lowest level.

When only two levels of nozzles are used, the top level can be desirably located in a zone in the tower where temperatures range from 165° to 185° F.

When a third level is to be used, it should preferably be spaced substantially equidistant the top level and the bottom level.

In FIG. **1**, a variation of the spacing of the spray nozzles is depicted by positioning them adjacent to the wall of the spray-drying chamber. A feedline **36** is indicated passing slurry to such nozzles. In such a position, care needs to be exercised that the spray from the nozzles is directed into the drying zone to avoid sticking to the vertical wall of the chamber.

In FIG. **2**, taken along **2**—**2** of FIG. **1**, the substantially uniform spacing of atomizing nozzles **20** is illustrated. These nozzles **20** are seen to be part of a manifold ring **42** leading to feedline **19**. It is important to space the spray nozzles throughout the tower in such a position that they are not too close to the chamber wall **39** or too close to the low-pressure vortex tube, **38**. If freshly sprayed slurry contacts the wall, it can tend to stick to the wall and build up large deposits. These must be removed with difficulty and they can obstruct the desirable gas flow patterns which the method and apparatus are designed to achieve.

In FIG. **2**, the plenum is indicated as **22** and the conveyor, **26** leads away from the tower.

The following examples illustrate the present invention. Variations and modifications can be made in the examples without deviating from the practices taught and contemplated by the present invention. Data is presented in tabular form

between two-level and three-level embodiments of this invention and comparisons made with ordinary single-level operation.

EXAMPLE I

A synthetic detergent slurry of the following approximate composition was prepared.

	Parts
Sodium tallow alkyl sulfate	9.2
Sodium dodecyl alkyl benzene sulfonate	7.6
Sodium sulfate	13.0
C_{12}—C_{14} amide ($RC(=O)—NH_2$)	1.1
$CNAE_6$—(condensate of coconut alcohol and 6 moles of ethylene oxide)	0.6
Sodium silicate	7.0
Sodium tripolyphosphate (STP)	34.6
Sodium nitrilotriacetate (NTA)	12.4
Water	40.8
Minors (CMC and brighteners)	1.0

The slurry was spray-dried under the following conditions with these results:

Spray nozzle arrangement	3–8′ 4–22′ 4–35′	5–8′ 5–35′	10–8′
Final product moisture, percent	8.5	8.5	7.0
Product rate, lb./hour	59,000	56,000	53,000
Product density, oz./100 in.^{3}	16½	16⅝	16¾
Percent on 14-mesh screen, percent	1.7	2.3	5.5
Coarse recycle level (observation)	Light	Moderate	Heavy.
Injection air level, approximate, percent	60 (54 s.c.f.m.)	58 (52 s.c.f.m.)	60 (54 s.c.f.m.)
Tower inlet air temperature, ° F	660	650	655
Tower exhaust air temperature, ° F	185	186	190
Production limitation	(1)	(1)	(2)
High-pressure pump pressure, p.s.i.	1,000	1,000	1,050

[1] Unable to convey product away from tower fast enough.
[2] Coarse recycle levels too high to handle.

The nozzles were uniformly spaced at each level. The changes in rate, density, coarse recycle amounts are all significant.

This example produced an excellent detergent product having reduced phosphate level. It demonstrates the efficacy of a mixed anionic-active system used with a builder mixture of STP and NTA.

EXAMPLE II

A synthetic detergent slurry was prepared similar to example I but without NTA·Na_3. Instead the STP level was increased to 47 parts. Spray-drying was performed using the following conditions.

Spray nozzle arrangement	3–8′ 3–22′ 4–35′	5–8′ 5–35′	9–8′
Final product moisture, percent	12.3	12.7	12.0
Product rate, lb./hour	63,500	62,000	58,000
Product density, oz./100 in.^{3}	16¾	17¼	17⅜
Percent on 14-mesh	1.6	3.1	4.8
Coarse recycle level	Moderate	Heavy	(1)
Injection air level, approximate percent	68 (61 s.c.f.m.)	70 (63 s.c.f.m.)	94 (85 s.c.f.m.)
Tower inlet air temperature, ° F	675	677	678
Tower exhaust air temperature, ° F	179	180	190
Production limitation	(2)	(3)	(4)
High-pressure pump pressure, p.s.i.	975	975	1,000

[1] Very heavy.
[2] Inability to pump detergent slurry faster.
[3] Coarse recycle levels too high to handle. Density high.
[4] Coarse recycle levels too high to handle. Product density high.

EXAMPLE III

A synthetic detergent slurry of the following composition was prepared:

Sodium dodecyl alkyl benzene sulfonate Sodium sulfate	12.7 parts
Tallow fatty acid	11.5 parts
Hardened marine fatty acid	1.5 parts
Sodium silicate	0.5 parts
STP	9.8 parts
NTA·Na_3	38.4 parts
Minors (CMC and brighteners)	10.3 parts
Water	1.0 parts
	36.7 parts

Spray-drying was performed under the following conditions with these results:

Spary nozzle arrangement	2 at 8′ 3 at 20′ 3 at 32′	8 at 8′
Product moisture, percent	8.8	9.4
Product rate, lb./hour	59,600	49,700
Product density, oz./100 in.^{3}	16¼	16⅝
Percent on 14-mesh	5.5	6.8
Coarse recycle level (measured) lb./hour	2,880	4,680
Injection air level, approximate, percent	29 (29 s.c.f.m.)	30 (30 s.c.f.m.)
Tower inlet air temperature, ° F	675	670
Tower exhaust air temperature, ° F	170	181
Product limitation	(1)	(2)
High-pressure pump pressure, p.s.i.	1,000	1,050

[1] Inability to pump more material to tower.
[2] Product sticky, coarse recycle too high to handle.

This example also demonstrates multilevel effectiveness in rates, density, coarse recycle, etc. over a single-level system.

EXAMPLE IV

Sodium dodecyl alkyl benzene sulfonate Sodium sulfate	16.8 parts
Sodium silicate	13.1 parts
STP	7.0 parts
NTA·Na_3	33.6 parts
Minors (CMC and brighteners)	12.5 parts
Water	1.1 parts
	36.0 parts

Spray-dried under the following conditions with these results:

Spray nozzle arrangement	3–8′ 3–20′ 3–32′	8–8′
Product moisture, percent	10.1	10.2
Product rate, lb./hour	53,600	44,300
Product density, oz./100 in.^{3}	16½	16½
Percent on 14-mesh screen	4.6	5.4
Coarse recycle level, lb./hour	2,940	3,123
Fine recycle (from exhaust system), lb./hour	565	1,680
Tower inlet air temperature, ° F	680	672
Tower exhaust air temperature, ° F	172	178
Production limitation	(1)	(2)
High-pressure pump pressure, p.s.i.	1,000	1,000

[1] Drying capacity of the spray tower (inlet temperature at maximum).
[2] Product sticky, coarse recycle became excessive when rate increased.

The decreased amount of fines produced with the three-level system of the present invention is very marked.

In this example the STP or the NTA can be replaced with an equal amount by weight of sodium citrate, sodium mellitate, sodium oxydiacetate, sodium oxydisuccinate with satisfactory results.

In each of these examples, the production of fines was reduced by approximately 50 percent. The product was free-flowing, uniformly sized granules.

Additional tests were performed convincingly showing the disadvantages of spraying fresh particles too close to the chamber wall or into the vortex zone of decreased pressure.

EXAMPLE V

The following slurry was prepared:

Tridecyl benzene sulfonate	9.7 parts
Condensation product of coconut alcohol and 6 moles of ethylene oxide 6)	3.3 parts
Sodium tripolyphosphate (STP)	4.8 parts
Sodium nitrilotriacetate ($NTA\text{-}Na_3$)	25.0 parts
Sodium silicate solids	10.6 parts
Sodium sulfate	28.1 parts
Hardened marine fatty acid	0.5 parts
Tallow fatty acid	1.5 parts
Minors (CMC and brighteners)	1.0 parts
Water	35.2 parts

Spray-drying was done with the following different nozzle configurations with the results indicated:

Nozzle Arrangement	3 Levels	Ordinary-1 level
	3 at 8 feet 2 at 22 feet 3 at 35 feet	7 at one 8 foot level
Production rate	48,000 lbs./hr.	35,000 lbs./hr.
Percent on 14 mesh screen	4.0	6.4
Density oz./100 $in.^3$	16–16¼	16½–17½
Product moisture	7.5%	5.5%
Product feel	Good	Sticky
% STP as hexahydrate by DSC	47%	0%
% STP species:		
STP	6.67	4.97
Pyrophosphate	4.73	5.41
Ortho	1.06	1.29
Trimeta	0.15	0.09

These show that: (1) up to 30 percent gain in tower rate is provided by the three-level nozzle arrangement; (2) drying conditions are less severe and product quality is guarded in that the STP hexahydrate is not broken down as much when multilevel nozzles are used; (3) processing is controllable.

The hexahydrate is primarily formed in the slurry prior to spraying into the drying tower. Therefore, the lower hexahydrate level in the product made with nozzles all at one level indicates more severe drying conditions. The analysis of phosphate species in the product substantiate this in that the higher levels of pyro- and orthophosphates in the product with single-level nozzles would result from overdrying the product (phosphate reversion).

Noteworthy also is that this formulation with all the nozzles at one level could not be produced at a higher rate because the product became too sticky to handle. The three-level operation was not rate limited, and was essentially free of stickiness.

In each of the foregoing examples, the lowest level was positioned at a point below a 190° F. isotherm and above a boiling point isotherm. The amount sprayed through each nozzle was approximately the same. The percentage sprayed into each zone is readily ascertainable by calculation. The amount sprayed into the lowest level was always in the range of 30 to 80 percent of the slurry produced.

In order to practice the present invention to produce synthetic detergent granules having increased density,

With the present invention it is possible to prepare synthetic detergent compositions of varied formulations.

The organic detergent can be selected from well-known classes of synthetic detergents including anionic, nonionic, ampholytic and zwitterionic detergents. These are illustrated by the following listed materials.

A. Anionic Soap and Nonsoap Synthetic Detergents

This class of detergents includes ordinary alkali metal soaps such as the sodium, potassium, ammonium and alkylolammonium salts of higher fatty acids containing from about eight to about 24 carbon atoms and preferably from about 10 to about 20 carbon atoms. Suitable fatty acids can be obtained from natural sources such as, for instance, from plant or animal esters (e.g., palm oil, coconut oil, babassu oil, soybean oil, castor oil, tallow, whale and fish oils, grease, lard, and mixtures thereof). The fatty acids also can be synthetically prepared (e.g., by the oxidation of petroleum, or by hydrogenation of carbon monoxide by the Fischer-Tropsch process). Resin acids are suitable such as rosin and those resin acids in tall oil. Napthenic acids are also suitable. Sodium and potassium soaps can be made by direct saponification of the fats and oils or by the neutralization of the free fatty acids which are prepared in a separate manufacturing process. Particularly useful are the sodium and potassium salts of the mixtures of fatty acids derived from coconut oil and tallow, i.e., sodium or potassium tallow and coconut soap.

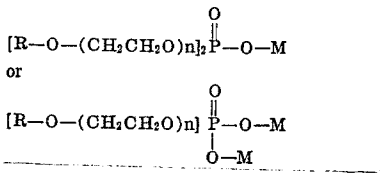
This class of detergents also includes water-soluble salts, particularly the alkali metal salts of organic sulfuric reaction products having in their molecular structure an alkyl radical containing from about eight to about 22 carbon atoms and a sulfonic acid or sulfuric acid ester radical. (Included in the term alkyl is the alkyl portion of higher acyl radicals.) Examples of this group of synthetic detergents which form a part of the preferred built detergent compositions of the present invention are the sodium or potassium alkyl sulfates, especially those obtained by sulfating the higher alcohols (C_8–C_{18} carbon atoms) produced by reducing the glycerides of tallow or coconut oil; sodium or potassium alkyl benzene sulfonates, in which the alkyl group contains from about nine to about 15 carbon atoms, in straight chain or branched-chain configuration, e.g., those of the type described in U.S. Pat. Nos. 2,220,099 and 2,477,383 (especially valuable are linear straight chain alkyl benzene sulfonates in which the average of the alkyl groups is about 13 carbon atoms abbreviated hereinafter as C_{13}LAS); sodium alkyl glyceryl ether sulfonates, especially those ethers of higher alcohols derived from tallow and coconut oil; sodium coconut oil fatty acid monoglyceride sulfonates and sulfates; sodium or potassium salts of sulfuric acid esters of the reaction product of 1 mole of a higher fatty alcohol (e.g., tallow or coconut oil alcohols) and about 1 to 6 moles of ethylene oxide; sodium or potassium salts of alkyl phenol ethylene oxide ether sulfate with about 1 to about 10 units of ethylene oxide per molecule and in which the alkyl radicals contain about eight to about 12 carbon atoms.

Additional examples of anionic nonsoap synthetic detergents which come within the terms of the present invention are the reaction product of fatty acids esterified with isethionic acid and neutralized with sodium hydroxide where, for example, the fatty acids are derived from coconut oil; sodium or potassium salts of fatty acid amide of methyl tauride in which the fatty acids, for example, are derived from coconut oil. Other anionic synthetic detergents of this variety are set forth in U.S. Pat. Nos. 2,486,921; 2,486,922; and 2,396,278.

Still other anionic synthetic detergents include the class designated as succinamates. This class includes such surface-active agents as disodium N-octadecylsulfo succinamate; tetrasodium N-(1,2-dicarboxyethyl)-N-octadecyl-sulfo-succinamate; diamyl ester of sodium sulfosuccinic acid; dihexyl ester of sodium sulfosuccinic acid; dioctyl ester of sodium sulfosuccinic acid.

Anionic phosphate surfactants are also useful in the present invention. These are surface-active materials having substantial detergent capability in which the anionic solubilizing group connecting hydrophobic moieties is an oxy acid of phosphorus. The more common solubilizing groups, of course, are $-SO_4H$, $-SO_3H$, and $-CO_2H$. Alkyl phosphate esters such as $(R{-}O)_2PO_2H$ and $ROPO_3H_2$ in which R represents an alkyl chain containing from about eight to about 20 carbon atoms are useful.

These esters can be modified by including in the molecule from one to about 40 alkylene oxide units, e.g., ethylene oxide units. Formulas for these modified phosphate anionic detergents are



in which R represents an alkyl group containing from about eight to 20 carbon atoms, or an alkylphenyl group in which the alkyl group contains from about eight to 20 carbon atoms, and M represents a soluble cation such as hydrogen, sodium, potassium, ammonium or substituted ammonium; and in which *n* is an integer from 1 to about 40.

A specific anionic detergent which has also been found excellent for use in the present invention is described more fully in the U.S. Pat. No. 3,332,880 of Phillip F. Pflaumer and Adriaan Kessler, issued July 25, 1967, titled Detergent Composition. This detergent comprises by weight from about 30 to about 70 percent of component A, from about 20 to about 70 percent of component B, and from about 2 to about 15 percent of component C, wherein:

a. said component A is a mixture of double-bond positional isomers of water-soluble salts of alkene-1-sulfonic acids containing from about 10 to about 24 carbon atoms, said mixture of positional isomers including about 10 to about 25 percent of an alpha-beta unsaturated isomer, about 30 to about 70 percent of a beta-gamma unsaturated isomer, about 5 to about 25 percent of a gamma-delta unsaturated isomer, and about 5 to about 10 percent of a delta-epsilon unsaturated isomer;

b. said component B is a mixture of water-soluble salts of bifunctionally substituted sulfur-containing saturated aliphatic compounds containing from about 10 to about 24 carbon atoms, the functional units being hydroxy and sulfonate radicals with the sulfonate radical always being on the terminal carbon and the hydroxyl radical being attached to a carbon atom at least two carbon atoms removed from the terminal carbon atom, at least 90 percent of the hydroxy radical substitutions being in the 3, 4, and 5 positions; and

c. said component C is a mixture comprising from about 30–95 percent water-soluble salts of alkene disulfonates containing from about 10 to about 24 carbon atoms, and from about 5 to about 70 percent water-soluble salts of hydroxy disulfonates containing from about 10 to about 24 carbon atoms, said alkene disulfonates containing a sulfonate group attached to a terminal carbon atom and a second sulfonate group attached to an internal carbon atom not more than about six carbon atoms removed from said terminal carbon atom, the alkene double bond being distributed between the terminal carbon atom and about the seventh carbon atom, said hydroxy disulfonates being saturated aliphatic compounds having a sulfonate radical attached to a terminal carbon, a second sulfonate group attached to an internal carbon atom not more than about six carbon atoms removed from said terminal carbon atom, and a hydroxy group attached to a carbon atom which is not more than about four carbon atoms removed from the site of attachment of said second sulfonate group.

B. Nonionic Synthetic Detergents

Nonionic synthetic detergents may be broadly defined as compounds produced by the condensation of alkylene oxide groups (hydrophilic in nature) with an organic hydrophobic compound, which may be aliphatic or alkyl aromatic in nature. The length of the hydrophilic or polyoxyalkylene radical which is condensed with any particular hydrophobic group can be readily adjusted to yield a water-soluble compound having the desired degree of balance between hydrophilic and hydrophobic elements.

For example, a well-known class of nonionic synthetic detergents is made available on the market under the trade name of "Pluronic." These compounds are formed by condensing ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The hydrophobic portion of the molecule which, of course, exhibits water insolubility, has a molecular weight of from about 1,500 to 1,800. The addition of polyoxyethylene radicals to this hydrophobic portion tends to increase the water solubility of the molecule as a whole and the liquid character of the product is retained up to the point where polyoxyethylene content is about 50 percent of the total weight of the condensation product.

Other suitable nonionic synthetic detergents include:

1. The polyethylene oxide condensates of alkyl phenols, e.g., the condensation products of alkyl phenols having an alkyl group containing from about six to 12 carbon atoms in either a straight chain or branched-chain configuration, with ethylene oxide, the said ethylene oxide being present in amounts equal to 5 to 25 moles of ethylene oxide per mole of alkyl phenol. The alkyl substituent in such compounds may be derived from polymerized propylene, diisobutylene, octene, or nonene, for example.

2. Those derived from the condensation of ethylene oxide with the product resulting from the reaction of propylene oxide and ethylene diamine. For example, compounds containing from about 40 percent to about 80 percent polyoxyethylene by weight and having a molecular weight of from about 5,000 to about 11,000 resulting from the reaction of ethylene oxide groups with a hydrophobic base constituted of the reaction product of ethylene diamine and excess propylene oxide, said base having a molecular weight of the order of 2,500 and 3,000, are satisfactory.

3. The condensation product of aliphatic alcohols having from eight to 22 carbon atoms, in either straight chain or branched-chain configuration, with ethylene oxide, e.g., a coconut alcohol-ethylene oxide condensate having from 5 to 30 moles of ethylene oxide per mole of coconut alcohol, the coconut alcohol fraction having from 10 to 14 carbon atoms.

4. Nonionic detergents include nonyl phenol condensed with either about 10 or about 30 moles of ethylene oxide per mole of phenol and the condensation products of coconut alcohol with an average of either about 5.5 or about 15 moles of ethylene oxide per mole of alcohol and the condensation product of about 15 moles of ethylene oxide with 1 mole of tridecanol.

Other examples include dodecylphenol condensed with 12 moles of ethylene oxide per mole of phenol; dinonylphenol condensed with 15 moles of ethylene oxide per mole of phenol; dodecyl mercaptan condensed with 10 moles of ethylene oxide per mole of mercaptan; bis-(N-2-hydroxyethyl) lauramid; nonyl phenol condensed with 20 moles of ethylene oxide per mole of nonyl phenol; myristyl alcohol condensed with 10 moles of ethylene oxide per mole of myristyl alcohol; lauramide condensed with 15 moles of ethylene oxide per mole of lauramide; and di-isooctylphenol condensed with 15 moles of ethylene oxide.

5. A detergent having the formula $R^1R^2R^3N\rightarrow O$ (amine oxide detergent) wherein R^1 is an alkyl group containing from about 10 to about 28 carbon atoms, from zero to about two hydroxy groups and from zero to about five ether linkages, there being at least one moiety of R^1 which is an alkyl group containing from about 10 to about 18 carbon atoms and zero ether linkages, and each R^2 and R^3 are selected from the group consisting of alkyl radicals and hydroxyalkyl radicals containing from one to about three carbon atoms;

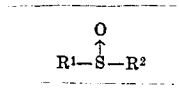
Specific examples of amine oxide detergents include: dimethyldodecylamine oxide, dimethyltetradecylamine oxide, ethylmethyltetradecylamine oxide, cetyldimethylamine oxide, dimethylstearylamine oxide, cetylethylpropylamine oxide, diethyldodecylamine oxide, diethyltetradecylamine oxide, dipropyldodecylamine oxide, bis-(2-hydroxyethyl)dodecylamine oxide, bis-(2-hydroxyethyl)-3-dodecoxy-1-hydroxypropylamine oxide, (2-hydroxypropyl)methyltetradecylamine oxide, dimethyloleyamine oxide, dimethyl-(2-hydroxydodecyl)amine oxide, and the corresponding

decyl, hexadecyl and octadecyl homologs of the the above compounds.

6. A detergent having the formula $R^1R^2R^3P\rightarrow O$ (phosphine oxide detergent) wherein R' is an alkyl group containing from about 10 to about 28 carbon atoms, from zero to about two hydroxy groups and from zero to about five ether linkages, there being at least one moiety of R' which is an alkyl group containing from about 10 to about 18 carbon atoms and zero ether linkages, and each of R^2 and R^3 are selected from the group consisting of alkyl radicals and hydroxyalkyl radicals containing from one to about three carbon atoms.

Specific examples of the phosphine oxide detergents include: dimethyldodecylphosphine oxide, dimethyltetradecylphosphine oxide, ethylmethyltetradecylphosphine oxide, cetyldimethylphosphine oxide, dimethylstearylphosphine oxide, cetylethylpropylphosphine oxide, diethyldodecylphosphine oxide, diethyltetradecylphosphine oxide, dipropyldodecylphosphine oxide, bis-(hydroxymethyl)dodecylphosphine oxide, bis-(2-hydroxyethyl)-dodecylphosphine oxide, (2-hydroxypropyl)methyltetradecylphosphine oxide, dimethyloleylphosphine oxide, and dimethyl-(2-hydroxydodecyl)phosphine oxide and the corresponding decyl, hexadecyl, and octadecyl homologs of the above compounds.

7. A detergent having the formula



(sulfoxide detergent) wherein R^1 is an alkyl radical containing from about 10 to about 28 carbon atoms, from zero to about five ether linkages and from zero to about two hydroxyl substituents at least one moiety of R^1 being an alkyl radical containing zero ether linkages and containing from about 10 to about 18 carbon atoms, and wherein R^2 is an alkyl radical containing from one to three carbon atoms and from one to two hydroxyl groups: octadecyl methyl sulfoxide, dodecyl methyl sulfoxide, tetradecyl methyl sulfoxide, 3-hydroxytridecyl methyl sulfoxide, 3-methoxytridecyl methyl sulfoxide, 3-hydroxy-4-dodecoxybutyl methyl sulfoxide, octadecyl 2-hydroxyethyl sulfoxide, dodecylethyl sulfoxide.

C. Ampholytic Synthetic Detergents

Ampholytic synthetic detergents can be broadly described as derivatives of aliphatic or aliphatic derivatives of heterocyclic secondary and tertiary amines, in which the aliphatic radical may be straight chain or branched and wherein one of the aliphatic substituents contains from about eight to 18 carbon atoms and at least one contains an anionic water-solubilizing group, e.g., carboxy, sulfo, sulfato, phosphato, or phosphono. Examples of compounds falling within this definition are sodium 3-(dodecylamino)-propionate, sodium 3-(dodecylamino)propane-1-sulfonate, sodium 2-(dodecylamino)ethyl sulfate, sodium 2-(dimethylamino)octadecanoate, disodium 3-(N-carboxymethyl-dodecylamino)-propane-1-sulfonate, disodium 2-(oleyamino)ethyl phosphate, disodium 3-(N-methylhexadecylamino)propyl-1-phosphonate, disodium octadecyl-iminodiacetate, sodium 1-carboxymethyl-2-undecylimidazole, disodium 2-[N-(2-hydroxyethyl)octadecylamino]N,N-bis-(2-hydroxyethyl)-2-sulfato-3-dodecoxypropylamine.

D. Zwitterionic Synthetic Detergents

Zwitterionic synthetic detergents can be broadly described as derivatives of aliphatic quaternary ammonium and phosphonium or tertiary sulfonium compounds, in which the cationic atom may be part of a heterocyclic ring, and in which the aliphatic radical may be straight chain or branched, and wherein one of the aliphatic substituents contains from about three to 18 carbon atoms, and at least one aliphatic substituent contains an anionic water-solubilizing group, e.g., carboxy, sulfo, sulfato, phosphato, or phosphono. Examples of compounds falling within this definition are 3-(N,N-dimethyl-N-hexadecyl-ammonio)-2-hydroxypropane-1-sulfonate, 3-N,N-dimethyl-N-hexadecylammonio)-propane-1-sulfonate, 2-(N,N-dimethyl-N-dodecylammonio)acetate, 3-(N,N-dimethyl N-dodecylammonio)propionate, 2-(N,N-dimethyl-N-octadecylammonio)-ethyl sulfate, 2-(trimethylammonio)ethyl dodecyl-phosphonate, ethyl 3-(N,N-dimethyl-N-dodecylammonio)-propylphosphonate, 3-(P,P-dimethyl-P-dodecylphosphonio)-propane-1-sulfonate, 2-(S-methyl-S-tert.-hexadecyl-sulfonio)ethane-1-sulfonate, 3-(S-methyl-S-dodecylsulfonio)-propionate, sodium 2-(N,N-dimethyl-N-dodecylamminio)ethyl phosphonate, 4-(S-methyl-S-tetradecylsulfonio)butyrate, 1-(2-hydroxyethyl)-2-undecylimidazolium-1-acetate, 2-(trimethylammonio)-octadecanote, and 3-(N,N-bis-(2-hydroxyethyl)-N-octodecylammonio)-2-hydroxypropane-1-sulfonate. Some of these detergents are described in the following U.S. Pat. Nos. 2,129,264; 2,178,353; 2,774,786; 2,813,898; and 2,828,332.

The builders can be any organic or inorganic builders.

Examples of suitable water-soluble, inorganic alkaline detergency builder salts are alkali metal carbonates, borates, phosphates, polyphosphates, bicarbonates, silicates and sulfates. Specific examples of such salts are sodium and potassium tetraborates, perborates, bicarbonates, carbonates, tripolyphosphates, pyrophosphates, orthophosphates and hexametaphosphates. Sodium sulfate, although not classed as an alkaline builder salt, is included in this category.

Examples of suitable organic alkaline detergency builder salts are: (1) water-soluble aminopolycarboxylates, e.g., sodium and potassium ethylenediaminetetraacetates, nitrilotriacetates and N-(2-hydroxyethyl)-nitrilodiacetates; (2) water-soluble salts of phytic acid, e.g., sodium and potassium phytates—see U.S. Pat. No. 2,739,942; (3) water-soluble, polyphosphonates, including specifically, sodium, potassium and lithium salts of ethane-1-hydroxy-1,1-diphosphonic acid, sodium, potassium and lithium salts of methylene diphosphonic acid, sodium, potassium and lithium salts of ethylene diphosphonic acid, and sodium, potassium and lithium salts of ethane-1,1,2-triphosphonic acid. Other examples include the alkali metal salts of ethane-2-carboxy-1,1-diphosphonic acid, hydroxymethanediphosphonic acid, carbonyldiphosphonic acid, ethane-1-hydroxy-1,1,2-triphosphonic acid, ethane-2-hydroxy-1,1,2-triphosphonic acid, propane-1,1,3,3-tetraphosphonic acid, propane-1,1,2,3-tetraphosphonic acid, and propane-1,2,2,3-tetraphosphonic acid; (4) water-soluble salts of polycarboxylate polymers and copolymers as described in the copending application of Francis L. Diehl, Ser. No. 268,359, filed Apr. 1, 1963, now U.S. Pat. No. 3,308,067. Specifically, a detergent builder material comprising a water-soluble salt of a polymeric aliphatic polycarboxylic acid having the following structural relationships as to the position of the carboxylate groups and possessing the following prescribed physical characteristics: (a) a minimum molecular weight of about 350 calculated as to the acid form; (b) an equivalent weight of about 50 to about 80 calculated as to acid form; (c) at least 45 mole percent of the monomeric species having at least two carboxyl radicals separated from each other by not more than two carbon atoms; (d) the site of attachment of the polymer chain of any carboxyl-containing radical being separated by not more than three carbon atoms along the polymer chain from the site of attachment of the next carboxyl-containing radical. Specific examples are polymers of itaconic acid, aconitic acid, maleic acid, mesaconic acid, fumaric acid, methylene malonic acid, and citraconic acid and copolymers with themselves and other compatible monomers such as ethylene; and (5) mixtures thereof.

Mixtures of organic and/or inorganic builders can be used and are generally desirable. One such mixture of builders is disclosed in the copending application of Burton H. Gedge, Ser. No. 398,705, filed Sept. 23, 1964, now U.S. Pat. No. 3,392,121, e.g., ternary mixtures of sodium tripolyphosphate, sodium nitrilotriacetate and trisodium ethane-1-hydroxy-1,1-diphosphonate. The above-described builders can also be utilized singly in this invention. Especially preFerred builders that

can be used singly or in combination in this invention include sodium perborate and sodium tripolyphosphate. Sodium tripolyphosphate and sodium perborate can be used in combination in a weight ratio range of from about 95:5 to about 50:50.

In addition, other builders can be used satisfactorily such as water-soluble salts of mellitic acid, citric acid, pyromellitic acid, benzene pentacarboxylic acid, oxydiacetic acid, oxydisuccinic acid.

All of the percentages and proportions used in describing the present invention are by weight unless otherwise specified.

The spray tower apparatus described herein is the subject of another commonly assigned U.S. Pat. application Ser. No. 60,012, filed July 31, 1970 entitled "Multilevel Spray Drying Apparatus," by Robert P. Davis, Michael S. Haines and John A. Sagel.

While the multilevel spray method of this invention gives overall finer product, the control of density is not lost in that the density of the individual sieve fractions is lighter. This is shown in the following particle size (sieve) distribution and the bulk density of the individual fractions:

Fractions Separated (Tyler Screen Mesh)	Percent of Product		Topped Bulk Density (g./cc.)	
	3 level	1 level	3 level	1 level
thru 14, on 20	10.7	18.2	0.296	0.327
thru 20, on 28	28.4	29.8	0.302	0.334
thru 28, on 35	24.6	20.0	0.322	0.345
thru 35, on 48	14.8	10.7	0.345	0.362
thru 48, on 65	10.5	7.4	0.371	0.394
thru 65, on 100	4.3	3.3	0.411	0.415

Nozzle Arrangement:

3 level=3 at 8 feet, 3 at 22 feet, 4 at 35 feet

1 level=9 at 8 feet

This data was obtained from product prepared in example II

The foregoing description of the present invention has been presented describing certain operable and preFerred embodiments. It is not intended that the invention should be so limited since variations and modifications thereof will be obvious to those skilled in the art, all of which are within the spirit and scope of this invention.

What is claimed is:

1. A continuous method for spray-drying large volumes of a synthetic detergent slurry in a spray-drying tower and producing a granular synthetic detergent composition having controlled density and uniform reduced particle size with minimum production of dust particles and other vapor effluents compromising the following steps:

2. preparing an aqueous synthetic detergent slurry having about 10–50 percent by weight water and the balance 50–90 percent solids content being comprised of at least one organic synthetic detergent, and at least one detergency builder selected from organic or inorganic builders or mixtures thereof;

2. establishing within the chamber of the spray tower (a) a cylindrically shaped drying zone with the axis of the chamber by passing heated drying air upwards through the chamber in a cyclonic motion and (b) a low-pressure zone comprising a concentric vortex tube which is formed along the axis of the chamber;

3. continuously spraying countercurrently from 30 to 80 percent of said synthetic detergent slurry directly into the cylindrically shaped drying zone at the point below a 190° F. isotherm and above a boiling point isotherm said spraying being achieved with atomizing nozzles substantially uniformly spaced in a horizontal plane through the cylindrical drying zone thereby providing that substantially each of the sprays disintegrates into particles within said cylindrical drying zone;

4. continuously spraying countercurrently the balance of said synthetic detergent slurry directly into the cylindrically shaped drying zone at a point above the 190° F. isotherm by means of at least one level of atomizing nozzles substantially uniformly spaced in a horizontal plane through the cylindrical drying zone, thereby providing that substantially each of the sprays disintegrates into particles within said cylindrical drying zone, whereby the only disintegrated particles entering the low-pressure vortex tube are those incidentally carried by the cyclonic motion of the drying gas.

2. A method according to claim 1 wherein in step 3 the amount sprayed is in the range of from 35 to 70 percent of said synthetic detergent slurry.

3. A method according to claim 1 wherein in step 4 the balance of said synthetic detergent slurry is sprayed into the cylindrically shaped drying zone by means of one level of uniformly spaced atomizing nozzles which are located at a point in the tower where the temperatures in the range of 165° to 185° F. obtain.

4. A method according to claim 3 wherein there is a third level of uniformly spaced atomizing nozzles located intermediate (a) the level of atomizing nozzles located in the tower where temperatures in the range of 165° to 185° F. obtain and (b) the level of atomizing nozzles located below the 190° F. isotherm.

5. A method according to claim 3 wherein the intermediate third level of atomizing nozzles is located substantially equidistant between the upper and lower levels.

6. A method according to claim 1 wherein the detergency builder is a phosphorus-free builder.

7. A method according to claim 6 wherein the detergency builder is a water-soluble salt of nitrilotriacetic acid.

8. A method according to claim 1 wherein the detergency builder is phosphorus-free and nitrogen-free.

9. A method according to claim 8 wherein the detergency builder is selected from a water-soluble salt of citric acid, mellitic acid or mixtures thereof.

10. A method according to claim 1 wherein the detergent is a nonsoap anionic synthetic detergent and the builder is one having reduced phosphate level comprising a mixture of sodium tripolyphosphate and sodium nitrilotriacetate in a molar ratio respectively of 3:1 to 1:6.

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