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PATENT SPECIFICATION

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NO DRAWINGS

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COMPLETE SPECIFICATION

Process for the preparation of Detergent Compositions

We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York, New York 10022, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the preparation of detergent compositions having a low bulk density.

The term "low bulk density" as used herein refers to compositions having an apparent density of less than 0.45 grams per cubic centimetre. By "apparent density" is meant the weight per unit volume of the material indicated by the context. Thus, in the case of freely flowing particulate products, the term refers to the untamped weight per unit volume of the particulate material as it is charged into a container.

The present invention provides a process for preparing a low bulk density detergent composition economically and in such a manner as to minimize the water content of the composition immediately prior to the drying step, thereby reducing the need for large quantities of drying heat, and large drying apparatus.

The present invention provides a process for the production of a solid detergent composition of low bulk density as hereinbefore defined comprising forming an aqueous slurry containing an organic detergent, adding to the slurry or forming therein an inorganic alkali metal or alkaline earth metal alkaline or neutral detergent builder salt, mixing the slurry in the presence of the builder salt and of a normally gaseous substance under conditions that impart a high shear and under superatmospheric pressure to disperse the gaseous substance uniformly therein, terminating the mixing, and reducing the pressure to atmospheric pressure whereby a low bulk density detergent product is formed.

The product, which may if desired be cooled after removal from the mixer, is a solid which granulates or is capable of being readily transformed into a particulate detergent product having a low bulk density.

It is advantageous, in preparing a low bulk density product which is capable of retaining such low bulk density throughout the steps of cooling and other subsequent treatment, to utilize a material which hydrates rapidly thereby imparting a relatively high structural strength to the detergent product. Suitable rapidly hydratable compounds include, for example, such materials as the alkali metal or alkaline earth metal trimetaphosphates which are capable of reaction with a strong base to form the corresponding alkali metal or alkaline earth metal tripolyphosphate hydrate.

In the practice of the invention, it is advantageous initially to form a premix of the slurry in the absence of any hydratable compound and then subsequently to incorporate the hydratable compound and the gaseous material with the premix immediately before introducing the premix into the mixer. It has been found that by virtue of this initial formation of a premix there can be obtained, in a manner and for reasons set forth more fully hereinafter, a product issuing from the mixer which has a higher solids content than could heretofore be obtained, thereby reducing the necessity of removing large quantities of water in forming the final product. The reduction in moisture content in forming the final product allows a reduction in capital and operating costs, such as by the use of smaller and more

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economical types of drying apparatus and correspondingly lower heat inputs.

This invention is particularly useful in the production of synthetic detergents from feeds of high solid content, from which feeds it has heretofore been difficult to obtain a product of sufficiently low bulk density. Thus, the invention facilitates the production of a desirable low bulk density product of low builder content even though the material during processing possesses a high solids content.

Detergents which may be used in the process of the present invention are water-soluble and organic in nature and in general should have foaming properties. Examples of suitable anionic detergents include water-soluble soaps and sulphonated synthetic detergents. The soaps are generally water-soluble salts of fatty acids (including rosin acids) which are derived usually from fats, oils and waxes of animal, vegetable or marine origin, e.g. tallow, coconut oil, tall oil and palm kernel oil. It is preferred to employ an alkyl aryl sulphonate such as an alkylbenzene sulphonate wherein the alkyl group has 8 to 16 carbon atoms. Suitable examples are sodium decyl, dodecyl and pentadecyl benzene sulphonates. Other suitable anionic detergents are surface-active sulphated or sulphonated aliphatic compounds, preferably having 8 to 22 carbon atoms, for example; long-chain pure or mixed alkyl sulphates (e.g. sodium lauryl sulphate); fatty acid ethanolamide sulphates (e.g. sodium coconut fatty acid ethanolamide sulphate); fatty acid amides of amino alkyl sulphonic acids (e.g. sodium lauric acid amide of taurine); and fatty acid esters of isethionic acid. These anionic detergents are used generally in the form of the water-soluble salts, such as the alkali metal (e.g. sodium, potassium) salts, though other water-soluble salts such as ammonium, alkylamine and alkaline earth metal salts may be used if desired, depending upon the particular detergent composition.

Also, the organic detergent may be, in whole or in part, a non-ionic detergent such as the non-ionic polyalkylene oxide condensates with an aliphatic or aromatic hydrophobic group. The hydrophobic organic group contains usually at least 8 carbon atoms and is condensed with at least 5 and usually up to 50 alkylene oxide groups. Examples are polyethylene oxide condensates with alkyl phenols having 6 to 20 carbon atoms in the alkyl group; polyethylene oxide esters of fatty acids such as tall oil acids or lauric acid with 16 to 20 ethylene oxide groups per molecule; polyethylene oxide condensates of aliphatic alcohols such as lauryl, myristyl, oleyl or stearyl alcohol with 6 to 30 moles of ethylene oxide per mole of the alcohol; and polyoxyethylene oxide condensates of fatty acid amides such as coconut fatty acid amide with 10 to 50 moles of ethylene oxide per mole of the amide. Water-soluble polyoxyethylene condensates with

hydrophobic polyoxypropylene glycols may be employed also. Suitable non-ionic detergents may be, for example, the condensate of ethylene oxide with polypropylene glycol, which condensate contains 80% ethylene oxide and has a molecular weight of about 1700, and iso-octyl phenoxy polyoxyethylene ethanol having about 8.5 ethanoxo groups per molecule.

A cationic surface-active agent may be incorporated in the product also. It may be admixed in powdered or liquid form with the other ingredients in any suitable manner. Where it is desired to use a cationic agent, it is preferred to admix it in minor proportion with an acidic ingredient and coat this mixture with a coating agent. Suitable cationic detergents are alkyl quaternary ammonium compounds such as cetyl quaternary ammonium salts. Specific examples of such materials are cetyl trimethyl ammonium chloride and cetyl pyridinium chloride. Similarly, ampholytic detergents such as salts of the N-alkyl compounds of beta amino propionic acid wherein the alkyl group is derived from a fatty acid such as a mixture of coconut oil fatty acids may be employed in compatible amounts. An example of such an ampholytic detergent is sodium dodecyl beta-alanine.

The detergent may in whole or in part be a fatty acid soap such as kettle soap. The presence of kettle soap facilitates the processing by imparting more rapid setting and aeration characteristics to the slurry. The soap itself lends some structural strength to the product and thereby reduces breakdown of the product particles. Suitable soaps include the water-soluble alkali metal, amine, and ammonium salts of fatty acids, such as those containing from 10 to 18 carbon atoms, e.g. sodium laurate, sodium myristate, potassium palmitate, triethanolamine stearate, and mixtures thereof with each other and with soaps of unsaturated fatty acids such as sodium oleate (e.g. the sodium soap of tallow fatty acids and the sodium soap of an 85:15 mixture of tallow and coconut oil fatty acids).

A suitable range of proportions of the organic detergent is from 2% to 65% of the finished product, and preferably 10% to 40% where the detergent is a synthetic material and 20 to 65% where a soap is the active material.

It is desirable to include in the composition a small amount of water-soluble hydrotropic alkyl aryl sulphonate to aid in the successful entrainment of gas in the necessary quantity and finely divided form in the slurries employed in carrying out the process. As much as 20% of this material can be used; however, it is usually desirable to include not more than 6% of the hydrotropic alkyl aryl sulphonate, while up to 2% is preferred. The hydrotropic alkyl aryl sulphonates used may be water-soluble hydrotropic agents characterized by a hydrophile-lipophile balance such that they are

highly water-soluble (more so, for example, than related organic detergents), and while exhibiting considerable surface activity, are substantially non-detergent in nature. Examples of suitable hydrotropes are sodium toluene sulphonate, sodium xylene sulphonate, sodium dibutyl naphthalene sulphonate, sodium (mono-) dodecyl oxydibenzene disulphonate, and the corresponding potassium and lithium salts and mixtures thereof, including commercially available isomeric mixtures. Preferably the alkyl substituent contains from 1 to 6 carbon atoms for each sulphonate group in the molecule, and the aryl nucleus desirably is benzene or naphthalene. The hydrotropic agent may be singly or plurally sulphonated or alkylated, or both. Although reference has been made to alkali metal salts of these compounds, other water-soluble salts such as the alkaline earth metal, e.g. magnesium, salts and ammonium and substituted ammonium, e.g. the triethanolamine, salts may be used totally or partially in lieu thereof.

In operation, the hydrotropic alkyl aryl sulphonate may be added to the slurry as such, or it may be added in the form of the sulphonic acid and neutralized in the presence of other constituents. Likewise, the hydrotropic alkyl aryl sulphonic acid may be prepared by sulphonation of the appropriate hydrocarbon in the presence of other constituents if desired, for example, by sulphonation with the same sulphonating agent used to prepare other sulphated sulphonated constituents of the composition, such as an alkyl aryl sulphonate detergent. Such co-sulphonation may be either simultaneous or tandem as appropriate, it being preferred to sulphonate the hydrotropic alkyl aryl hydrocarbon as the last material to be sulphonated in the latter case.

One form of this invention involves the provision of a material capable of reaction with water to produce a water-soluble hydrate, preferably a polyphosphate hydrate. The formation of a hydrate of this type removes free water from the slurry and imparts structural strength to the finished product, thereby reducing or eliminating break-down of the product, for example, when under the weight of superimposed material. Suitable hydratable compounds include alkali metal and alkaline earth metal phosphates such as pentasodium tripolyphosphate, both Forms I and II, tetrasodium pyrophosphate, potassium pyrophosphate, trisodium orthophosphate, and sodium trimetaphosphate. Also suitable are such hydratable compounds as sodium carbonate, sodium metasilicate, magnesium sulphate, sodium tetraborate and mixtures thereof. The hydratable material is used in particulate form. At least 90% of the particles will advantageously have a diameter smaller than 150 microns, and preferably at least 97% of the particles have a diameter smaller than 150

microns while at least 85% thereof have a diameter smaller than 75 microns.

In determining the most suitable hydratable compound to be used, it is preferred to use a rapidly hydratable compound. The faster the rate of hydration, the more quickly the structural strength will be increased, thereby allowing the foraminous system to become self-supporting. Accordingly, it is preferred that a compound be chosen which is capable of substantial hydration in less than 30 minutes at the temperatures of the process. Suitably, a period of 1 to 10 minutes is satisfactory, but a period of 2 to 5 minutes is preferred. A preferred hydratable compound is sodium trimetaphosphate, which, in the presence of a strong base, rapidly reacts to form sodium tripolyphosphate hexahydrate. Similarly, other alkali metal trimetaphosphates can be used to produce alkali metal tripolyphosphate hydrates. The hydratable material may be used in amounts of 20 to 80%. The amount of hydratable material will advantageously be in the range 30 to 50%. Preferably, the hydratable material is an alkali metal trimetaphosphate which is utilized in the range of 30 to 50%.

The conversion of trimetaphosphate to tripolyphosphate hydrate requires reaction with a strong base such as the alkali metal carbonates, silicates, oxides, hydroxides, orthophosphates and alkaline earth metal oxides and hydroxides. The base should be sufficiently strong to give a pH in excess of 10.2 at 25°C. when 1% by weight is dissolved in distilled water. The more desirable strong bases are the alkali metal hydroxides, carbonates and silicates; sodium and potassium hydroxides are the preferred bases.

The strong base should be used in amounts sufficient to cause the desired conversion of the alkali metal trimetaphosphate into the corresponding alkali metal tripolyphosphate hydrate. Generally, the ratio of strong base to trimetaphosphate will be between 1.4:1 to 10:1 and preferably from 2:1 to 6:1 mole equivalents. The exact amount will depend upon the particular base and trimetaphosphate to be used.

It may be desirable to include in the composition certain compounds in addition to those set forth above, which compounds may be useful for various purposes such as fillers, e.g. sodium sulphate, anti-redeposition agents, e.g. sodium carboxymethyl cellulose, buffers, brighteners, colourants and stabilizers.

The water content of the slurry during the high shear mixing step is generally less than that at which the material can readily be pumped or otherwise caused to flow through a conduit without some difficulty. This may be accomplished by initially forming a premix which contains all or a major portion of the water and a portion only of the solids, and commingling the remainder of the solids therewith just before the high shear mixing. The

total solids content of the slurry employed in carrying out the process of the present invention at the time it is mixed in the high shear mixer may vary usually from 70 to 85% of the total composition, depending upon the other constituents present and the processing conditions employed. A preferred range for the solids content is 75 to 80%. In any event, enough water must be used so that the slurry, after the intensive mixing step, is of such viscosity and fluidity that it forms a uniform paste having a consistency which permits it to swell or expand but which prevents rise of gas through the paste and therefore substantially preserves a structure of thoroughly dispersed discrete bubbles, preventing substantial coalescence of the gaseous bubbles or loss of gas from the system.

The gaseous material which is intimately admixed with the slurry to form bubbles therein may be any suitable normally gaseous material such as air, nitrogen, oxygen or carbon dioxide. It is preferred to use air.

The process produces a product of low apparent density and a high rate and degree of solubility in water. When granulated, the particulate products produced exhibit little tendency towards dust formation or caking during transportation and/or storage, and have excellent odour characteristics compared with products made from comparable materials in accordance with presently employed commercial procedures. Moreover, in contrast to many relatively high temperature heat-dried detergent compositions, the present products have a negligible content of products of thermal degradation of organic or inorganic constituents and thus, when made from water-soluble materials, are completely water-soluble.

For continuous operation, wherein a premix is formed and continuously fed to a mixer, and an additive such as trimetaphosphate in combination with a strong base is continuously added just prior to the high shear mixing step set forth in detail hereinafter, it is particularly desirable to provide a continuous metered flow of the premix and additive streams to allow continuous and accurate proportioning of the components. In embodiments of the process wherein detergent material of high solids content is employed, which material is difficultly flowable, it is important to be able rapidly to adjust the relative proportions of the component streams to obtain a uniform product. For this purpose use may be made of apparatus as described and claimed in the complete specification of British Patent Application No. 24663/66 (Serial No. 1,151,766) from which the present application has been divided out.

As already mentioned, certain materials should be added immediately prior to the high shear mixing step. This is especially important where a degradable or hydratable material such as sodium tripolyphosphate or sodium

trimetaphosphate is used and reactions immediately begin to occur. For example, sodium tripolyphosphate is subject both to hydration and hydrolysis. When hydration occurs water is removed from the mixture. When working at high solids concentration, hydration causes the slurry to become even less fluid so that it cannot be caused to flow or adequately mixed. Other reactions such as hydrolysis cause degradation of the material, resulting in an undesirable final product. Accordingly, it is necessary to minimize both the hold-up time and the mixing time. It is therefore preferred that the premix slurry and the additive material remain for as short a period of time as possible in feeding devices which supply them to the high shear mixer. The time after all of the components of the final product have been commingled and before the high shear mixing step is begun is suitably 5 seconds or even less. The throughput time in the high shear mixer will advantageously be less than 30 seconds, and preferably between 5 to 20 seconds.

The gaseous material is injected into the mixture before it enters the high shear mixer.

Where an alkali metal trimetaphosphate is to be added, the reaction between the hydratable material and the water present in the premix slurry is not initiated until the trimetaphosphate is brought into the process of caustic alkali. In such instance, the material does not form substantial amounts of hydrate until the caustic alkali is added. Thus, all or a part of the trimetaphosphate may be incorporated into the premix slurry earlier than would otherwise be possible. The exact point of time of addition of the hydratable material to the premix slurry depends upon many variables and operating parameters, such as the rate at which hydration occurs, the temperatures involved, the solids content and the relative amounts of the compounds.

The mixer for intimately and continuously mixing the components and evenly dispersing the gaseous material therein must impart a high shear to the mixture. Suitable mixers are generally relatively small in capacity so that at any particular moment only a small quantity of slurry is being worked upon thereby allowing a major portion of the power input to be used directly on the slurry without becoming dissipated in an attempt to mix large quantities of material at one time. Thus, the mixer will advantageously have a capacity of less than one gallon, whereby material can be passed through the mixer in less than 30 seconds while being subjected to extremely high shear for this short period of time. The mixer may be of any suitable high shear type and, for example may comprise a mixing chamber having two stators and a rotor adapted to rotate between the stators. The internal faces of both of the stators and both faces of the rotor may be provided with concentric rows

of blades arranged so that the blades of the rotor mesh closely but out of contact with the blades of the stators. The slurry may advantageously pass from an inlet at the centre of one stator, between the blades of that stator and the rotor, across the rotor, and between the blades of the other stator and the rotor, and then to an exit at the far side of the mixer. In passing through the mixer the slurry and the gaseous material are torn, stretched and cut by the blades into numerous streams, and minute bubbles of the gaseous material are evenly distributed throughout the slurry. Other types of mixer may be used so long as the

mixer is capable of imparting large quantities of energy and high shear rates to the slurry whereby a major proportion (i.e. more than 50 percent) of the gaseous material is broken into discrete bubbles having an average diameter of less than 0.085 millimetre and preferably less than 0.054 millimetre.

The forces applied to the mixture vary with the equipment used and the operating parameters of the process. By "high shear" is meant a shearing action of the type set forth wherein there is a "Shear Factor (F)" as defined below in excess of 5 and preferably in excess of 6.5.

$$\text{Shear Factor (F)} = \frac{2 \times 10^{-6} \pi r RT}{dC}$$

wherein:

r = the radius in inches of the mixer rotor or its equivalent;

R = the number of revolutions per minute of the mixer rotor or its equivalent;

T = the rate of feed in pound per hour of the material to be sheared;

d = the clearance in inches between the blades on the rotor and those on the stator or their equivalents;
and

C = the volumetric capacity of the mixer in cubic inches.

A tube may be provided at the outlet of the mixer for transfer of the intimately mixed material from the mixer on to a conveyor. The tube may also serve to adjust the internal pressure in the mixer and for this purpose may be varied in length and diameter. For example, by increasing the length or decreasing the diameter of the tube, the pressure within the mixer can be increased. It is desirable to use a pressure-controlling member of this type instead of a throttle valve, to avoid a sudden drop in pressure which may cause instability of the product. The length and diameter of the tube may be selected to maintain the mixer pressure within the range 20 to 100 pounds per square inch gauge, preferably within the range 40 to 65 pounds per square inch gauge.

The conveyor may comprise a continuously moving belt whereby the product is rapidly removed thereby eliminating piling up of product to an undesirable height which might tend to break down the structure of the product, causing the final bulk density to be higher than desired. It may in some cases be desirable to maintain the temperature of the product on the conveyor at a predetermined

level for a predetermined time, in which instance the conveyor may be provided with means to heat and/or cool it. The product may be discharged from the conveyor into a dryer, e.g. a fluidized bed heating chamber, a rotary kiln, a continuous belt which passes through a heating tunnel, a spray drying tower or a vertical shaft heating chamber. For reasons of economy, however, it is preferred that a tumbling drum type of dryer be used. Especially economical drying is made possible by virtue of the low free moisture content of the product. Perfume may be added to the product at any suitable time, as at the end of the drying step.

The following examples illustrate the invention. Unless otherwise indicated, all parts, proportions and percentages in the examples and elsewhere are by weight, and all inorganic salts are anhydrous.

EXAMPLE I.

An open throat "progressing cavity" feeder was continuously fed with two streams of material. One stream consisted of a slurry made from the following materials:

	Parts by weight
Water	15.6
50° Bé caustic soda solution	11.2
Sodium toluene sulphonate (commercial grade)	3.0
Tridecylbenzene sulphonic acid (96% sulphonic acid, 2% sulphuric acid, 1% free oil, 1% moisture)	35.2
Sodium sulphate	13.7
Sodium carboxymethyl cellulose (74% active ingredient, 26% inert solids)	1.6
Sodium silicate (44.1% solids having an $\text{Na}_2\text{O}:\text{SiO}_2$ ratio of 1:2.0)	19.7

The slurry constituting the first stream was maintained at a temperature of about 57°C. and was delivered to a feeder from a metering device at the rate of 304 pounds per hour. The second stream consisted of Form II powdered pentasodium tripolyphosphate delivered from a metering device at a rate of 161 pounds per hour. Form II pentasodium tripolyphosphate is a phosphate which during manufacture is calcined at a low temperature as compared to Form I which is calcined at a higher temperature. Form I pentasodium tripolyphosphate hydrates more rapidly than Form II thereof. The Form II pentasodium tripolyphosphate used in this example was in the form of particles of which 86% were less than 75 microns in diameter, and 11% had a diameter between 75 and 150 microns.

The mixture of powder and slurry was subjected to some preliminary mixing before discharge from the feeder, and the material discharged was a mixture of powder and slurry in a substantially constant ratio. The solids content of the material at this point was 77%.

The feed was pumped directly to a continuous high shear mixer. The hold-up time was about 30 seconds. Immediately upstream of the mixer a metered stream of compressed air was introduced, and the mixture then

entered the mixer within 5 seconds. This air amounted to about 4.0 standard cubic feet of air per hour. The mixer was operated at a rotor speed of 400 RPM. A 14-inch length of $\frac{1}{4}$ inch inside diameter tubing on the mixer discharge maintained an operating pressure of about 60 pounds per square inch gauge at the inlet of the mixer, and provided for a gradual diminution of the pressure on the material being processed.

The material discharged from the high shear mixer was a uniformly mixed and aerated paste with a density of about 0.44 grams per cubic centimetre at a temperature of about 65°C. It was fed into a series of tubs, where it was allowed to remain in a quiescent state at room temperature for about 48 hours. During this period it solidified. The product was removed from the tubs and passed through a rotating cage mill to reduce all lumps to small particles and was then screened through a sieve having square apertures each of 2 millimetres a side. The resulting powder had an apparent density of 0.38 grams per cubic centimetre and a moisture content of about 22%, and was dried to 18% total moisture in a moving current of warm (60°C.) air. This product had a composition approximately as follows:

	Percent
Tridecylbenzene sodium sulphonate	25
Pentasodium tripolyphosphate	37
Sodium toluene sulphonate	2
Sodium silicate	6
Sodium carboxymethyl cellulose	0.8
Sodium sulphate	11
Water (essentially all molecularly bound by the phosphate and other hydratable salts)	18
Other matter	0.2

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EXAMPLES II—VIII.

A series of experiments was carried out under varying operating conditions and with a composition having the same formulation of starting materials as that described in Example I, except as otherwise indicated. The operating conditions are set forth in Table I below:

TABLE I

Ex.	Temperature of Premix Slurry (1)	Feed Rate (2)	Solids Content (percent) (3)	Air (Standard cubic feet per hour) (4)	Rotor Speed of Mixer (RPM) (5)	Pressure (6)	Temp. (7)	Prod. Dens. (8)	Prod.* Mois. (per-cent)	Shear Factor	STS (9)
II.	125	360	75	3.1	400	35	135	0.37	23.7	7.6	2
III.	120	360	77	0.9	400	40	150	0.34	15.8	7.7	6
IV.	125	350	75	0.5	400	29	145	0.306	22.4	7.4	6
V.	160	350	75	6.3	400	39	152	0.34	22.8	7.5	2
VI.	135	459	75.5	4.6	400	42	137	0.367	26.0	9.8	2
VII.	125	348	76	4.0	400	53	140	0.371	23.6	7.4	2
VIII.	135	355	75.5	3.4	400	46	135	0.362	23.0	7.5	2
Run A	120	200	75	6.6	375	35	140			4.0	2
Run B	135	366	80.6	3.4	400	40	145			7.8	2

(1) This temperature was measured in degrees Fahrenheit.

(2) The feed rate was calculated in pounds per hour.

(3) The solids content was calculated as including all components and without any hydration.

(4) Standard cubic feet was calculated at a pressure of 760 millimetres mercury and a temperature of 0 degrees Centigrade.

(5) The rotor had a diameter of 8 inches. The mixer had a capacity of 79 cubic inches, and the clearance between the blades of rotor and stator was 0.60 inch.

(6) The pressure was measured in pounds per square inch gauge at the inlet of the high shear mixer.

(7) This temperature was measured in degrees Fahrenheit at the inlet of the high shear mixer.

(8) The product density was measured in grams per cubic centimetre.

(9) The amount of sodium toluene sulphate in the final product expressed as percent by weight.

*Product Moisture

50 45 40 35 30 25 20 15 10 5

In Examples IV and V Form I pentasodium tripolyphosphate was substituted for the Form II pentasodium tripolyphosphate used in most of the other examples. The Form I compound hydrates more rapidly than Form II pentasodium tripolyphosphate. A comparison of the product of Example IV with that of the otherwise similar Example III in which Form II pentasodium tripolyphosphate was used showed the improvement possible through use of a more rapidly hydratable material to impart structural strength to the product as it leaves the high shear mixing device.

As a comparison to the examples of the invention set forth above, Run A was performed wherein the entire formulation was made up in a batching device and the hydratable

material, Form II pentasodium tripolyphosphate, was included in this batch instead of being added later in the processing as in the above examples. The completely formulated material was fed by the feeder to the high shear mixer. During the course of a half hour run, the mix became too thick to pump due to substantial hydration of the hydratable material.

Run B illustrates another comparison run wherein insufficient (only 4%) hydratable material was used. The formulation differed from that used for Examples I—VIII. In Run B, the feeder was continuously fed with a first stream comprising a slurry made from the following materials:

	Percent
Water	27.77
50° Bé caustic soda solution	12.60
Sodium toluene sulphonate (powdered commercial grade)	3.41
Tridecylbenzene sulphonic acid	49.67
Form II Pentasodium tripolyphosphate	6.55

The slurry constituting the first stream was maintained at a temperature of about 155°F. and was delivered to the feeder at the rate of 188 parts by weight per hour. A second stream comprising 178 parts by weight per hour of anhydrous sodium sulphate as filler was delivered to the feeder and therein combined with the initial slurry.

The feed was pumped to a high shear mixer under approximately the same conditions as those described in Example I and about 3.4 standard cubic feet per hour of air was introduced. A pressure of about 40 pounds per square inch gauge was maintained at the inlet to the mixer. The material discharged from the mixer was a wet paste and failed to form an acceptable product even though a lesser

amount of water was used than in the examples set forth above. The cause of this is believed to be lack of sufficient hydratable material to bind the water, thereby providing inadequate structural strength. In addition it was difficult to feed the slurry due to the initial incorporation in the slurry of the hydratable material.

EXAMPLES IX—XV.

In this series of examples, the hydratable material was sodium trimetaphosphate and the examples were carried out in the following manner:

The feeder was continuously fed with two metered streams of material. A first stream comprised a slurry of the following:

(7) This temperature was measured in grams per cubic centimetre.
(8) The product density was measured in the final product expressed as percent by weight.
(9) The amount of sodium toluene sulphonate in the final product expressed as percent by weight.

*Product Moisture

	Parts by weight
Sodium toluene sulphonate	3.1
Sodium tridecylbenzene sulphonate (54% solids, 46% water; 86% active ingredient, 14% inert ingredient)	31.0
Sodium silicate solution (43.5% solids, 56.5% water)	14.0
Kettle soap (sodium salt of a mixture of fatty acids derived from tallow and coconut oils in the weight ratio of 80:20 respectively)	7.0
Sodium carboxymethyl cellulose (74% active ingredient, 26% inert ingredient)	0.8
Powdered polyvinyl alcohol	0.3
Ultra-marine blue	0.3
Sodium sulphate powder	43.5

5 The premix slurry constituting the first stream was maintained at a temperature of about 150°F. and was delivered to the feeder at a rate of 250 pounds per hour. The solids content of the premix slurry for Examples IX—XV varied between 73.5% and 79.0%.
 10 A second stream comprising powdered sodium trimetaphosphate having an average particle size less than 75 microns was metered to the feeder at the rate of 154 pounds per hour. The feeder delivered the mixture directly to a continuous high shear mixer operating at a shear factor in excess of 5. Immediately upstream
 15 of the mixer, a 50° Bé caustic soda solution was metered in at the rate of 60 pounds per hour and air was metered in at the rate shown in Table II. The product resulting from each of these examples was a low bulk density
 20 detergent composition of uniform consistency

having density and moisture contents as shown in Table II. These products could be further dried in any suitable manner to an even lower moisture content.

The product densities shown in Table II illustrate an advantage of the present invention in that it permits the preparation of a product having a bulk density lower than 0.35 even though the processing conditions included the use of a solids content higher than 80%, and also notwithstanding the fact that the final product contained less than 35% of tripolyphosphate, which is a relatively low tripolyphosphate content for low bulk density detergent compositions. Illustrative of the low tripolyphosphate contents obtained are Examples IX and XI which had tripolyphosphate contents of 27.8 and 27.3 respectively.

TABLE II

Ex.	Temperature of Premix Slurry (1)	Feed Rate (2)	Solids Content percent (3)	Air (Standard cubic feet per hour) (4)	Rotor Speed of Mixer (PRM) (5)	Pressure (6)	Temp. (7)	Prod. Dens. (8)	Prod.* Mois. (per-cent) (9)	Shear Factor (9)	STS (9)
IX.	150	464	83.5	0.5	800	21	190	0.15	15.0	19.6	2.0
X.	150	464	83.5	2.2	800	23	190	0.18	15.2	19.6	2.0
XI.	150	464	83.5	0.5	800	25	190	0.38	16.0	19.6	0.0
XII.	150	464	80.4	2.2	800	21	190	0.33	17.0	19.6	0.0
XIII.	190	464	80.0	2.2	400	23	190	0.27	18.5	9.8	0.0
XIV.	190	464	80.8	0.5	800	20	190	0.28	18.1	19.6	0.0
XV.	190	464	80.8	0.5	400	23	190	0.18	17.0	9.8	0.25

(1) This temperature was measured in degrees Fahrenheit.

(2) The feed rate was calculated in pounds per hour.

(3) The solids content was calculated as including all components and without any hydration.

(4) Standard cubic feet was calculated at a pressure of 760 millimetres mercury and a temperature of 0 degrees Centigrade.

(5) The rotor had a diameter of 8 inches. The mixer had a capacity of 79 cubic inches, and the clearance between the blades of rotor and stator was 0.060 inch.

(6) The pressure was measured in pounds per square inch gauge at the inlet of the high shear mixer.

(7) This temperature was measured in degrees Fahrenheit at the outlet of the high shear mixer.

(8) The product density was measured in grams per cubic centimetre.

(9) The amount of sodium toluene sulphonate in the final product expressed as percent by weight.

*Product Moisture.



WHAT WE CLAIM IS:—

1. A continuous process for the production of a detergent compositions of low bulk density as hereinbefore defined, which comprises forming an aqueous slurry containing an organic detergent, adding to the slurry or forming therein an inorganic alkali metal or alkaline earth metal alkaline or neutral detergent builder salt, mixing the slurry in the presence of the builder salt and of a normally gaseous substance under conditions that impart a high shear and under superatmospheric pressure to disperse the gaseous substance uniformly therein, terminating the mixing, and reducing the pressure at atmospheric pressure whereby a low bulk density detergent product is formed.
2. A process as claimed in Claim 1 in which the builder salt comprises an alkali metal trimetaphosphate.
3. A process as claimed in Claim 1 or Claim 2 in which the builder salt is formed *in situ* by commingling with the slurry a caustic alkali and a substance capable of reaction with water in the presence of the caustic alkali to produce a water-soluble alkali metal polyphosphate hydrate.
4. A process as claimed in Claim 3 in which the slurry and builder salt are intimately mixed, immediately after the said commingling, at a shear factor, as hereinbefore defined, in excess of 5.
5. A process as claimed in Claim 1 in which the builder salt is a hydratable material or a substance capable of forming a hydratable material.
6. A process as claimed in Claim 5 wherein the hydratable material or substance capable of forming the same is selected from alkali metal and alkaline earth metal phosphates, sodium carbonate, sodium silicate, magnesium sulphate, and sodium tetraborate.
7. A process as claimed in Claim 5 or Claim 6 wherein the hydratable material or substance capable of forming the same is a particulate solid having a particle size smaller than 150 microns.
8. A process as claimed in any of Claims 5 to 7 wherein the hydratable material or substance capable of forming the same is an alkali metal trimetaphosphate.
9. A process as claimed in any of the preceding claims wherein the slurry has such a

high solids content as to be difficultly flowable and wherein the relative proportions of the slurry and the builder salt are maintained substantially constant.

10. A process as claimed in any of the preceding Claims wherein the slurry is continuously and automatically metered to a point where the builder salt is (or the substances capable of forming the same *in situ* are) added in an automatically controlled and continuously metered amount.

11. A process as claimed in any of the preceding claims wherein the solids content of the slurry during the high shear mixing step is in excess of 70%; and the slurry is mixed in the presence of the normally gaseous substance for a period of up to 30 seconds and at a pressure in excess of 20 pounds per square inch gauge.

12. A process as claimed in any of the preceding claims wherein the mixing step disperses the normally gaseous substance throughout the slurry as discrete bubbles the majority of which have an average diameter less than 0.054 mm.

13. A process as claimed in any of the preceding claims wherein the mixing step is carried out at a shear factor, as hereinbefore defined, in excess of 5.

14. A process as claimed in any of the preceding claims wherein the high shear mixing step is performed under a pressure in excess of 40 pounds per square inch gauge.

15. A process as claimed in any of the preceding claims wherein the solids content of the slurry during the high shear mixing step is in excess of 75%.

16. A process as claimed in any of the preceding claims wherein the low bulk density detergent product is dried.

17. A continuous process for the production of a detergent composition of low bulk density, as hereinbefore defined, substantially as described in any of the Examples.

18. Detergent compositions of low bulk density, as hereinbefore defined, which have been produced by a process as claimed in any of the preceding claims.

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ERRATUM

SPECIFICATION No. 1,151,767

Page 8, Footnote 5, for "0.60" read "0.060"

THE PATENT OFFICE
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Int. Cl.

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