(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 929 653 B1

(12) EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the grant of the patent:
23.04.2003 Bulletin 2003/17

(21) Application number: **97931058.8**

(22) Date of filing: **05.06.1997**

(51) Int Cl.[7]: **C11D 11/00**, C11D 17/06

(86) International application number:
PCT/US97/09794

(87) International publication number:
WO 98/014556 (09.04.1998 Gazette 1998/14)

(54) **PROCESS FOR MAKING A DETERGENT COMPOSITION BY NON-TOWER PROCESS**

VERFAHREN ZUR HERSTELLUNG VON EINER WASCHMITTELZUSAMMENSETZUNG NACH OHNE-TURM-VERFAHREN

PROCEDE DE PRODUCTION D'UNE COMPOSITION DETERGENTE SANS L'UTILISATION D'UNE TOUR

(84) Designated Contracting States:
AT BE CH DE DK ES FI FR GB GR IE IT LI LU NL PT SE

(30) Priority: **04.10.1996 WOPCT/US96/15881**

(43) Date of publication of application:
21.07.1999 Bulletin 1999/29

(73) Proprietor: **THE PROCTER & GAMBLE COMPANY**
Cincinnati, Ohio 45202 (US)

(72) Inventors:
- **BEIMESCH, Wayne, Edward**
  Covington, KY 41015 (US)
- **ROMO, Jose Francisco Correa**
  Naucalpan, Edo. de Mexico 53100 (MX)
- **ROGERS, Steven, Barrett**
  Cincinnati, OH 45247 (US)

(74) Representative: **Peet, Jillian Wendy et al**
Procter & Gamble Technical Centres Limited,
Whitley Road,
Longbenton
Newcastle upon Tyne NE12 9TS (GB)

(56) References cited:

EP-A- 0 663 439	**WO-A-92/01036**
WO-A-93/25378	**WO-A-95/12659**
WO-A-96/09370	**GB-A- 2 209 172**
US-A- 5 489 392	**US-A- 5 554 587**

Remarks:
The file contains technical information submitted after the application was filed and not included in this specification

EP 0 929 653 B1

Description

FIELD OF THE INVENTION

[0001] The present invention generally relates to a non-tower process for producing a particulate detergent composition. More particularly, the invention is directed to a continuous process during which detergent agglomerates are produced by feeding a surfactant and coating materials into a series of mixers. The process produces a free flowing, detergent composition whose density can be adjusted for wide range of consumer needs, and which can be commercially sold.

BACKGROUND OF THE INVENTION

[0002] Recently, there has been considerable interest within the detergent industry for laundry detergents which are "compact" and therefore, have low dosage volumes. To facilitate production of these so-called low dosage detergents, many attempts have been made to produce high bulk density detergents, for example with a density of 600 g/l or higher. The low dosage detergents are currently in high demand as they conserve resources and can be sold in small packages which are more convenient for consumers. However, the extent to which modern detergent products need to be "compact" in nature remains unsettled. In fact, many consumers, especially in developing countries, continue to prefer a higher dosage levels in their respective laundering operations.

[0003] Generally, there are two primary types of processes by which detergent granules or powders can be prepared. The first type of process involves spray-drying an aqueous detergent slurry in a spray-drying tower to produce highly porous detergent granules (e.g., tower process for low density detergent compositions). In the second type of process, the various detergent components are dry mixed after which they are agglomerated with a binder such as a nonionic or anionic surfactant, to produce high density detergent compositions (e.g., agglomeration process for high density detergent compositions). In the above two processes, the important factors which govern the density of the resulting detergent granules are the shape, porosity and particle size distribution of said granules, the density of the various starting materials, the shape of the various starting materials, and their respective chemical composition.

[0004] There have been many attempts in the art for providing processes which increase the density of detergent granules or powders. Particular attention has been given to densification of spray-dried granules by post tower treatment. For example, one attempt involves a batch process in which spray-dried or granulated detergent powders containing sodium tripolyphosphate and sodium sulfate are densified and spheronized in a Marumerizer®. This apparatus comprises a substantially horizontal, roughened, rotatable table positioned within and at the base of a substantially vertical, smooth walled cylinder. This process, however, is essentially a batch process and is therefore less suitable for the large scale production of detergent powders. More recently, other attempts have been made to provide continuous processes for increasing the density of "post-tower" or spray dried detergent granules. Typically, such processes require a first apparatus which pulverizes or grinds the granules and a second apparatus which increases the density of the pulverized granules by agglomeration. While these processes achieve the desired increase in density by treating or densifying "post tower" or spray dried granules, they are limited in their ability to go higher in surfactant active level without subsequent coating step. In addition, treating or densifying by "post tower" is not favourable in terms of economics (high capital cost) and complexity of operation. Moreover, all of the aforementioned processes are directed primarily for densifying or otherwise processing spray dried granules. Currently, the relative amounts and types of materials subjected to spray drying processes in the production of detergent granules has been limited. For example, it has been difficult to attain high levels of surfactant in the resulting detergent composition, a feature which facilitates production of detergents in a more efficient manner. Thus, it would be desirable to have a process by which detergent compositions can be produced without having the limitations imposed by conventional spray drying techniques.

[0005] To that end, the art is also replete with disclosures of processes which entail agglomerating detergent compositions. For example, attempts have been made to agglomerate detergent builders by mixing zeolite and/or layered silicates in a mixer to form free flowing agglomerates. While such attempts suggest that their process can be used to produce detergent agglomerates, they do not provide a mechanism by which starting detergent materials in the form of pastes, liquids and dry materials can be effectively agglomerated into crisp, free flowing detergent agglomerates.

[0006] Accordingly, there remains a need in the art to have an agglomeration (non-tower) process for continuously producing a detergent composition having high density delivered directly from starting detergent ingredients, and preferably the density can be achieved by adjusting the process condition. Also, there remains a need for such a process which is more efficient, flexible and economical to facilitate large-scale production of detergents (1) for flexibility in the ultimate density of the final composition, and (2) for flexibility in terms of incorporating several different kinds of detergent ingredients (especially liquid ingredients) into the process.

[0007] The following references are directed to densifying spray-dried granules: Appel et al, U.S. Patent No. 5,133,924 (Lever); Bortolotti et al, U.S. Patent No. 5,160,657 (Lever); Johnson et al, British patent No. 1,517,713

(Unilever); and Curtis, European Patent Application 451,894.

[0008] The following references are directed to producing detergents by agglomeration: Beujean et al, Laid-open No.WO93/23,523 (Henkel), Lutz et al, U.S. Patent No. 4,992,079 (FMC Corporation); Porasik et al, U.S. Patent No. 4,427,417 (Korex); Beerse et al, U.S. Patent No. 5,108,646 (Procter & Gamble); Capeci et al, U.S. Patent No. 5,366,652 (Procter & Gamble); Hollingsworth et al, European Patent Application 351,937 (Unilever); Swatling et al, U.S. Patent No. 5,205,958; Dhalewadikar et al, Laid Open No.WO96/04359 (Unilever).

[0009] For example, the Laid-open No.WO93/23,523 (Henkel) describes the process comprising pre-agglomeration by a low speed mixer and further agglomeration step by high speed mixer for obtaining high density detergent composition with less than 25 wt% of the granules having a diameter over 2 mm. The U.S. Patent No. 4,427,417 (Korex) describes continuous process for agglomeration which reduces caking and oversized agglomerates.

[0010] US5554587 discloses a process for preparing detergent compositions comprising inputting air into a mixer during the agglomeration of a surfactant paste and other detergent ingredients, so that at least a minor amount of water from said surfactant paste is absorbed by said air.

[0011] WO9609370 discloses a process for preparing high density detergent compositions comprising the steps of mixing a surfactant paste and dry detergent ingredients in a mixer to form first agglomerates, conditioning said first agglomerates to obtain second agglomerates having a specific particle size, recycling any agglomerates not having the desired particle size for further agglomeration to obtain second agglomerates having the desired particle size, admixing detergent ingredients to said second agglomerates to form a high density detergent composition.

[0012] EP663439 discloses a process for making a detergent component which comprises the steps of making a detergent paste comprising water-soluble silicate salt and surfactant or water-soluble polymer, and dispersing said paste with a builder powder under pressure.

[0013] WO9325378 discloses a process for preparing detergent compositions comprising the steps of dispersing a surfactant paste through a powder stream, agglomerating the paste and powder in a mixer and drying or cooling the agglomerates.

[0014] WO9512659 addresses the problem of preparing low relative humidity detergents in elevated process temperatures and solves this problem by employing a pre-conditioned gas in the process conditions.

[0015] US5489392 discloses a process for preparing high density detergents comprising the steps of mixing a surfactant paste in a mixer to form agglomerates, screening said agglomerates to form a first agglomerate mixture, separating the agglomerates according to particle size and either grinding or recycling agglomerates for further agglomeration to obtain a final agglomerate mixture having the desired particle size, admixing detergent ingredients to the final agglomerate mixture to obtain a high density detergent composition.

[0016] WO9201036 discloses a process for producing surfactant granules comprising the steps of mixing a water-containing surfactant with a solid to form granules, drying the granules and recycling the dried granules as a portion of the solid.

[0017] It is also known to use fluidised bed for other purposes when manufacturing particulate detergents. For instance in GB-A-2,209,172 it is proposed to spray a liquid material on to fluidised particulate material comprising a builder and, in particular, it is proposed to utilise this for achieving in situ reaction in the fluid bed between fluidised particulate material comprising at least one alkaline builder support and liquid material which is sprayed on to the bed and which comprises at least one acid precursor of an anionic detergent active compound.

[0018] The process does not use the conventional spray drying towers currently which is limited in producing high surfactant loading compositions. In addition, the process of the present invention is more efficient, economical and flexible with regard to the variety of detergent compositions which can be produced in the process. Moreover, the process is more amenable to environmental concerns in that it does not use spray drying towers which typically emit particulates and volatile organic compounds into the atmosphere.

[0019] As used herein, the term "agglomerates" refers to particles formed by agglomerating raw materials with binder scuh as surfactants and or inorganic solutions / organic solvents and polymer solutions. As used herein, the term "granulating" refers to fluidising agglomerates thoroughly for producing free flowing, round shape granulated-agglomerates. As used herein, the term "mean residence time" refers to following definition:

$$\text{mean residence time (hr)} = \text{mass (kg)} / \text{flow throughput (kg/hr)}$$

[0020] All percentages used herein are expressed as "percent-by-weight" unless indicated otherwise. All ratios are weight ratios unless indicated otherwise. As used herein, "comprising" means that other steps and other ingredients which do not affect the result can be added. This term encompasses the terms "consisting of" and "consisting essentially of".

[0021] In accordance with one aspect of the invention, a process for preparing a granular detergent composition having a density at least about 600g/l is provided.

[0022] The process comprises the steps of:

(a) dispersing a surfactant, and coating the surfactant with fine powder having a diameter from 0.1 to 500 microns, in a mixer wherein conditions of the mixer include (i) from 2 to 50 seconds of mean residence time, (ii) from 4 to 25 m/s of tip speed, and (iii) from 0.15 to 7 kj/kg of energy condition, wherein agglomerates are formed; and

(b) granulating the agglomerates in fluidised bed apparatus while spraying into the bed droplets of liquid detergent material in an amount of up to 20% and wherein conditions of the fluidising apparatus include (i) from 1 to 10 minutes of mean residence time, (ii) from 100 to 300 mm of depth of unfluidised bed, (iii) not more than 50 micron of droplet spray size, (iv) from 175 to 250 mm of spray height, (v) from 0.2 to 1.4 m/s of fluidising velocity and (vi) from 12 to 100°C of bed temperature.

[0023] The invention results in the production of granular detergent compositions having a high density of at least about 600g/l.

<u>First Step [Step (a)]</u>

[0024] In the first step of the process, surfactant, i.e., one or more of aqueous and/or non-aqueous surfactant(s), which is/are in the form of powder, paste and/or liquid, and fine powder having a diameter from 0.1 to 500 microns, preferably from about 1 to about 100 microns are fed into a mixer, so as to make agglomerates. (The definition of the surfactants and the fine powder are described in detail hereinafter.) Optionally, an internal recycle stream of powder, having a diameter of about 0.1 to about 300 microns generated from fluidising apparatus which is described hereinafter in the second step, can be fed into the mixer in addition to the fine powder. The amount of such internal recycle stream of powder can be 0 to about 60 wt% of the final product from the process of the present invention.

[0025] In another embodiment of the invention, the surfactant(s) can be initially fed into a mixer or premixer (e.g., a conventional screw extruder or other similar mixer) prior to the above, after which the mixed detergent materials are fed into the first step mixer as described herein for agglomeration.

[0026] The mean residence time of the mixer is in range from about 2 to about 50 seconds and tip speed of the mixer is in range from about 4 m/s. to about 25 m/s, the energy per unit mass of the mixer (energy condition) is in the range from about 0.15 kj/kg to about 7 kj/kg, more preferably, the mean residence time of the mixer is in range from about 5 to about 30 seconds and tip speed of the mixer is in range from about 6 m/s to about 18 m/s, the energy per unit mass of the mixer (energy condition) is in range from about 0.3 kj/kg to about 4 kj/kg, and most preferably, the mean residence time of the mixer is in range from about 5 to about 20 seconds and tip speed of the mixer is in range from about 8 m/s to about 18 m/s, the energy per unit mas of the mixer (energy condition) is from about 0.3 kj/kg to about 4 kj/kg.

[0027] The examples of mixers for the first step can be any types of mixer known to the skilled in the art, as long as the mixer can maintain the above mentioned condition for the first step. An example can be Lodige CB Mixer manufacture by the Lodige company (Germany). As a result of the first step, resultant product (agglomerates having fine powder on the surface of the agglomerates) is then obtained.

<u>Second Step</u>

[0028] In the second step of the process, the resultant product of the first step (the agglomerates) is fed into a fluidised bed apparatus while spraying into the bed droplets of liquid detergent material in an amount of up to 20%, in order to enhance granulation for producing free flowing high density granules. In the second step, the resultant product from the first step is fluidised thoroughly so that the granules from the second step have a round shape. Optionally, about 0 to about 10%, more preferably about 2-5% of powder detergent materials of the kind used in the first step and/or other detergent ingredients can be added to the second step. The liquid detergent materials sprayed into the bed may be of the kind used in the first step and/or other detergent ingredients can be added to the step, for enhancing granulation and coating on the surface of the granules.

[0029] To achieve the density of at least about 600g/l, preferably more than 650g/l, condition of a fluidised apparatus is:

Mean residence time: from about 1 to about 10 minutes
Depth of unfluidised bed: from about 100 to about 300 mm
Droplet spray size: not more than about 50 micron
Spray height: from about 175 to about 250 mm
Fluidising velocity: from about 0.2 to about 1.4 m/s

Bed temperature: from about 12 to about 100°C, more preferably
Mean residence time: from about 2 to about 6 minutes
Depth of unfluidised bed: from about 100 to about 250mm
Droplet spray size: less than about 50 micron
Spray height: from about 175 to about 200 mm.
Fluidised velocity: from about 0.3 to about 1.0 m/s
Bed temperature: from about 12 to about 80°C.

[0030] The second step can utilise more than one fluidised apparatus (e.g., combining different kinds of fluidised apparatus such as fluid bed dryer and fluid bed cooler). If there is more than one fluidised apparatus, each may be operated under the same ranges of conditions, but the atomised liquid may be present only in one of the beds. If two different kinds of fluidised apparatus would be used, mean residence time of the second step in total can be from about 2 to about 20 minutes, more preferably, from about 2 to 12 minutes.

[0031] A coating agent to improve flowability and/or minimise over agglomeation of the detergent composition can be added in one or more of the following locations of the instant process: (1) the coating agent can be added directly after the fluid bed cooler or fluid bed dryer and/or (2) the coating agent may be added between the fluid bed dryer and the fluid bed cooler. The coating agent is preferably selected from the group consisting of aluminosilicates, silicates, carbonates and mixtures thereof. The coating agent not only enhances the free flowability of the resulting detergent composition which is desirable by consumers in that it permits easy scooping for detergent during use, but also serves to control agglomeration by preventing or minimizing over agglomeration. As those skilled in the art are well aware, over agglomeration can lead to very undesirable flow properties and aesthetics of the final detergent product.

Starting Detergent Materials

[0032] The total amount of the surfactants in products made by the present invention, which are included in the following detergent materials, finely atomized liquid and adjunct detergent ingredients is generally from about 5% to about 60%, more preferably from about 12% to about 40%, more preferably, from about 15 to about 35%, in percentage ranges. The surfactants which are included in the above can be from any part of the process of the present invention., e.g., from either one of the first step and/or the second step of the present invention.

Detergent Surfactant (Agueous /Non-agueous)

[0033] The amount of the surfactant of the present process can be from about 5% to about 60%, more preferably from about 12% to about 40%, more preferably, from about 15 to about 35%, in total amount of the final product obtained by the process of the present invention.

[0034] The surfactant of the present process, which is used as the above mentioned starting detergent materials in the first step, is in the form of powdered, pasted or liquid raw materials.

[0035] The surfactant itself is preferably selected from anionic, nonionic, zwitterionic, ampholytic and cationic classes and compatible mixtures thereof. Detergent surfactants useful herein are described in U.S. Patent 3,664,961, Norris, issued May 23, 1972, and in U.S. Patent 3,929,678, Laughlin et al., issued December 30, 1975. Useful cationic surfactants also include those described in U.S. Patent 4,222,905, Cockrell, issued September 16, 1980, and in U.S. Patent 4,239,659, Murphy, issued December 16, 1980. Of the surfactants, anionics and nonionics are preferred and anionics are most preferred.

[0036] Nonlimiting examples of the preferred anionic surfactants useful in the present invention include the conventional C_{11}-C_{18} alkyl benzene sulfonates ("LAS"), primary, branched-chain and random C_{10}-C_{20} alkyl sulfates ("AS"), the C_{10}-C_{18} secondary (2,3) alkyl sulfates of the formula $CH_3(CH_2)_x(CHOSO_3^-M^+)$ CH_3 and CH_3 $(CH_2)_y(CHOSO_3^-M^+)$ CH_2CH_3 where x and (y + 1) are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, and the C_{10}-C_{18} alkyl alkoxy sulfates ("AE_XS"; especially EO 1-7 ethoxy sulfates).

[0037] Useful anionic surfactants also include water-soluble salts of 2-acyloxy-alkane-1-sulfonic acids containing from about 2 to 9 carbon atoms in the acyl group and from about 9 to about 23 carbon atoms in the alkane moiety; water-soluble salts of olefin sulfonates containing from about 12 to 24 carbon atoms; and beta-alkyloxy alkane sulfonates containing from about 1 to 3 carbon atoms in the alkyl group and from about 8 to 20 carbon atoms in the alkane moiety.

[0038] Optionally, other exemplary surfactants useful in the paste of the invention include C_{10}-C_{18} alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the C_{10-18} glycerol ethers, the C_{10}-C_{18} alkyl polyglycosides and the corresponding sulfated polyglycosides, and C_{12}-C_{18} alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the C_{12}-C_{18} alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C_6-C_{12} alkyl phenol alkoxylates (especially ethoxylates and mixed ethoxy/propoxy), C_{10}-C_{18} amine oxides, and the like, can also be included in the overall compositions. The C_{10}-C_{18} N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C_{12}-C_{18} N-methylglucamides. See WO 9,206,154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C_{10}-C_{18} N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C_{12}-C_{18} glucamides can be used for low sudsing. C_{10}-C_{20} conventional soaps may also be used. If high sudsing is desired, the branched-chain C_{10}-C_{16} soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

[0039] Cationic surfactants can also be used as a detergent surfactant herein and suitable quaternary ammonium surfactants are selected from mono C_6-C_{16}, preferably C_6-C_{10} N-alkyl or alkenyl ammonium surfactants wherein remaining N positions are substituted by methyl, hydroxyethyl or hydroxypropyl groups.

[0040] Ampholytic surfactants can also be used as a detergent surfactant herein, which include aliphatic derivatives of heterocyclic secondary and tertiary amines; zwitterionic surfactants which include derivatives of aliphatic quaternary ammonium, phosphonium and sulfonium compounds; water-soluble salts of esters of alpha-sulfonated fatty acids; alkyl ether sulfates; water-soluble salts of olefin sulfonates; beta-alkyloxy alkane sulfonates; betaines having the formula $R(R^1)_2N^+R^2COO^-$, wherein R is a C_6-C_{18} hydrocarbyl group, preferably a C_{10}-C_{16} alkyl group or C_{10}-C_{16} acylamido alkyl group, each R^1 is typically C_1-C_3 alkyl, preferably methyl and R_2 is a C_1-C_5 hydrocarbyl group, preferably a C_1-C_3 alkylene group, more preferably a C_1-C_2 alkylene group. Examples of suitable betaines include coconut acylamidopropyldimethyl betaine; hexadecyl dimethyl betaine;

C_{12-14} acylamidopropylbetaine; C_{8-14} acylamidohexyldiethyl betaine;

4[C_{14-16} acylmethylamidodiethylammonio]-1-carboxybutane; C_{16-18} acylamidodimethylbetaine; C_{12-16} acylamidopentanediethylbetaine; and

[C_{12-16} acylmethylamidodimethylbetaine. Preferred betaines are C_{12-18} dimethyl-ammonio hexanoate and the C_{10-18} acylamidopropane (or ethane) dimethyl (or diethyl) betaines; and the sultaines having the formula $(R(R^1)_2N^+R^2SO_3^-$ wherein R is a C_6-C_{18} hydrocarbyl group, preferably a C_{10}-C_{16} alkyl group, more preferably a C_{12}-C_{13} alkyl group, each R^1 is typically C_1-C_3 alkyl, preferably methyl, and R^2 is a C_1-C_6 hydrocarbyl group, preferably a C_1-C_3 alkylene or, preferably, hydroxyalkylene group. Examples of suitable sultaines include C_{12}-C_{14} dimethylammonio-2-hydroxypropyl sulfonate, C_{12}-C_{14} amido propyl ammonio-2-hydroxypropyl sultaine, C_{12}-C_{14} dihydroxyethylammonio propane sulfonate, and C_{16-18} dimethylammonio hexane sulfonate, with C_{12-14} amido propyl ammonio-2-hydroxypropyl sultaine being preferred.

Fine Powder

[0041] The amount of the fine powder of the present process, which is used in the first step, can be from about 94% to 30%, preferably from 86% to 54%, in total amount of starting material for the first step . The starting fine powder of the present process preferably selected from the group consisting of ground soda ash, powdered sodium tripolyphosphate (STPP), hydrated tripolyphosphate, ground sodium sulphates, aluminosilicates, crystalline layered silicates, nitrilotriacetates (NTA), phosphates, precipitated silicates, polymers, carbonates, citrates, powdered surfactants (such as powdered alkane sulfonic acids) and internal recycle stream of powder occurring from the process of the present invention, wherein the average diameter of the powder is from 0.1 to 500 microns, preferably from 1 to 300 microns, more preferably from 5 to 100 microns. In the case of using hydrated STPP as the fine powder of the present invention, STPP which is hydrated to a level of not less than 50% is preferable. The aluminosilicate ion exchange materials used herein as a detergent builder preferably have both a high calcium ion exchange capacity and a high exchange rate. Without intending to be limited by theory, it is believed that such high calcium ion exchange rate and capacity are a function of several interrelated factors which derive from the method by which the aluminosilicate ion exchange material is produced. In that regard, the aluminosilicate ion exchange materials used herein are preferably produced in accordance with Corkill et al, U.S. Patent No. 4,605,509 (Procter & Gamble).

[0042] Preferably, the aluminosilicate ion exchange material is in "sodium" form since the potassium and hydrogen forms of the instant aluminosilicate do not exhibit as high of an exchange rate and capacity as provided by the sodium form. Additionally, the aluminosilicate ion exchange material preferably is in over dried form so as to facilitate production of crisp detergent agglomerates as described herein. The aluminosilicate ion exchange materials used herein preferably have particle size diameters which optimize their effectiveness as detergent builders. The term "particle size diameter" as used herein represents the average particle size diameter of a given aluminosilicate ion exchange material as determined by conventional analytical techniques, such as microscopic determination and scanning electron microscope (SEM). The preferred particle size diameter of the aluminosilicate is from about 0.1 micron to about 10 microns, more preferably from about 0.5 microns to about 9 microns. Most preferably, the particle size diameter is from about 1 microns to about 8 microns.

[0043] Preferably, the aluminosilicate ion exchange material has the formula

$$Na_z[(AlO_2)_z.(SiO_2)_y]xH_2O$$

wherein z and y are integers of at least 6, the molar ratio of z to y is from about 1 to about 5 and x is from about 10 to about 264. More preferably, the aluminosilicate has the formula

$$Na_{12}[(AlO_2)_{12}.(SiO_2)_{12}]xH_2O$$

wherein x is from about 20 to about 30, preferably about 27. These preferred aluminosilicates are available commercially, for example under designations Zeolite A, Zeolite B and Zeolite X. Alternatively, naturally-occurring or synthetically derived aluminosilicate ion exchange materials suitable for use herein can be made as described in Krummel et al, U.S. Patent No. 3,985,669, the disclosure of which is incorporated herein by reference.

[0044] The aluminosilicates used herein are further characterized by their ion exchange capacity which is at least about 200 mg equivalent of $CaCO_3$ hardness/gram, calculated on an anhydrous basis, and which is preferably in a range from about 300 to 352 mg equivalent of $CaCO_3$ hardness/gram.

Finely Atomized Liquid

[0045] The amount of the finely atomized liquid sprayed into the fluidised bed of the present process can be from about 1% to about 10% (active basis), preferably from 2% to about 6% (active basis) in total amount of the final product obtained by the process of the present invention. The finely atomized liquid of the present process can be selected from the group consisting of liquid silicate, anionic or cationic surfactants which are in liquid form, aqueous or non-aqueous polymer solutions, and mixtures thereof. Other optional examples for the finely atomized liquid of the present invention can be sodium carboxy methyl cellulose solution, polyethylene glycol (PEG), and solutions of dimethylene triamine pentamethyl phosphonic acid (DETMP),

[0046] The preferable examples of the anionic surfactant solutions which can be used as the finely atomized liquid in the present inventions are about 88 - 97% active HLAS, about 30 - 50% active NaLAS, about 28% active AE3S solution, about 40-50% active liquid silicate, and so on.

[0047] Cationic surfactants can also be used as finely atomized liquid herein and suitable quaternary ammonium surfactants are selected from mono C_6-C_{16}, preferably C_6-C_{10} N-alkyl or alkenyl ammonium surfactants wherein remaining N positions are substituted by methyl, hydroxyethyl or hydroxypropyl groups.

[0048] Preferable examples of the aqueous or non-aqueous polymer solutions which can be used as the finely atomized liquid in the present inventions are modified polyamines which comprise a polyamine backbone corresponding to the formula:

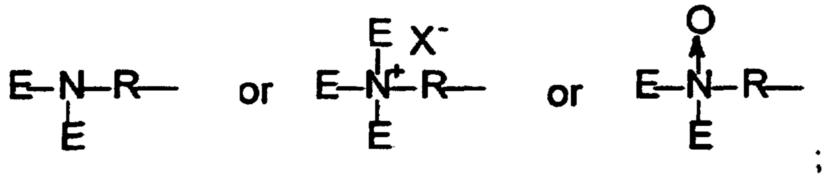
$$[H_2N-R]_{n+1}-[\overset{H}{N}-R]_m-[\overset{|}{N}-R]_n-NH_2$$

having a modified polyamine formula $V_{(n+1)}W_mY_nZ$ or a polyamine backbone corresponding to the formula:

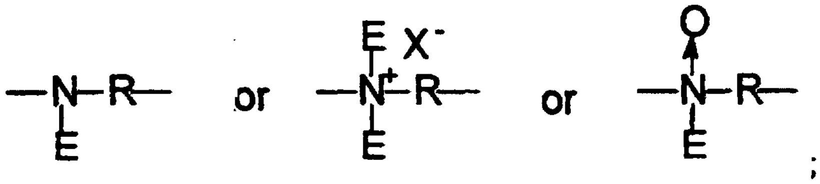
$$[H_2N-R]_{n-k+1}-[\overset{H}{N}-R]_m-[\overset{|}{N}-R]_n-[\overset{R}{N}-R]_k-NH_2$$

having a modified polyamine formula $V_{(n-k+1)}W_mY_nY'_kZ$, wherein k is less than or equal to n, said polyamine backbone prior to modification has a molecular weight greater than about 200 daltons, wherein

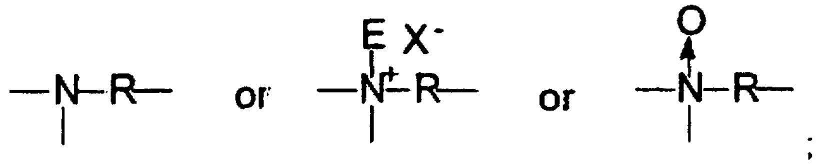
i) V units are terminal units having the formula:



ii) W units are backbone units having the formula:



iii) Y units are branching units having the formula:



and

iv) Z units are terminal units having the formula:

wherein backbone linking R units are selected from the group consisting of C_2-C_{12} alkylene, C_4-C_{12} alkenylene, C_3-C_{12} hydroxyalkylene, C_4-C_{12} dihydroxyalkylene, C_8-C_{12} dialkylarylene, $-(R^1O)_xR^1-$, $-(R^1O)_xR^5(OR^1)_x-$, $-(CH_2CH(OR^2)CH_2O)_z(R^1O)_yR^1(OCH_2CH(OR^2)CH_2)_w-$, $-C(O)(R^4)_rC(O)-$, $-CH_2CH(OR^2)CH_2-$, and mixtures thereof; wherein R^1 is C_2-C_6 alkylene and mixtures thereof; R^2 is hydrogen, $-(R^1O)_xB$, and mixtures thereof; R^3 is C_1-C_{18} alkyl, C_7-C_{12} arylalkyl, C_7-C_{12} alkyl substituted aryl, C_6-C_{12} aryl, and mixtures thereof; R^4 is C_1-C_{12} alkylene, C_4-C_{12} alkenylene, C_8-C_{12} arylalkylene, C_6-C_{10} arylene, and mixtures thereof; R^5 is C_1-C_{12} alkylene, C_3-C_{12} hydroxyalkylene, C_4-C_{12} dihydroxyalkylene, C_8-C_{12} dialkylarylene, $-C(O)-$, $-C(O)NHR^6NHC(O)-$, $-R^1(OR^1)-$, $-C(O)(R^4)_rC(O)-$, $-CH_2CH(OH)CH_2-$, $-CH_2CH(OH)CH_2O(R^1O)_yR^1OCH_2CH(OH)CH_2-$, and mixtures thereof; R^6 is C_2-C_{12} alkylene or C_6-C_{12} arylene; E units are selected from the group consisting of hydrogen, C_1-C_{22} alkyl, C_3-C_{22} alkenyl, C_7-C_{22} arylalkyl, C_2-C_{22} hydroxyalkyl, $-(CH_2)_pCO_2M$, $-(CH_2)_qSO_3M$, $-CH(CH_2CO_2M)CO_2M$, $-(CH_2)_pPO_3M$, $-(R^1O)_xB$, $-C(O)R^3$, and mixtures thereof; oxide; B is hydrogen, C_1-C_6 alkyl, $-(CH_2)_qSO_3M$, $-(CH_2)_pCO_2M$, $-(CH_2)_q(CHSO_3M)CH_2SO_3M$, $-(CH_2)_q-(CHSO_2M)CH_2SO_3M$, $-(CH_2)_pPO_3M$, $-PO_3M$, and mixtures thereof; M is hydrogen or a water soluble cation in sufficient amount to satisfy charge balance; X is a water soluble anion; m has the value from 4 to about 400; n has the value from 0 to about 200; p has the value from 1 to 6, q has the value from 0 to 6; r has the value of 0 or 1; w has the value 0 or 1; x has the value from 1 to 100; y has the value from 0 to 100; z has the value 0 or 1. One example of the most preferred polyethyleneimines would be a polyethyleneimine having a molecular weight of 1800 which is further modified by ethoxylation to a degree of approximately 7 ethyleneoxy residues per nitrogen (PEI 1800, E7). It is preferable for the above polymer solution to be pre-complex with anionic surfactant such as NaLAS.

[0049] Other preferable examples of the aqueous or non-aqueous polymer solutions which can be used as the finely atomized liquid in the present invention are polymeric polycarboxylate dispersants which can be prepared by polymerizing or copolymerizing suitable unsaturated monomers, preferably in their acid form. Unsaturated monomeric acids that can be polymerized to form suitable polymeric polycarboxylates include acrylic acid, maleic acid (or maleic anhydride), fumaric acid, itaconic acid, aconitic acid, mesaconic acid, citraconic acid and methylenemalonic acid. The presence in the polymeric polycarboxylates herein of monomeric segments, containing no carboxylate radicals such as vinylmethyl ether, styrene, ethylene, etc. is suitable provided that such segments do not constitute more than about 40% by weight of the polymer.

[0050] Homo-polymeric polycarboxylates which have molecular weights above 4000, such as described next are preferred. Particularly suitable homopolymeric polycarboxylates can be derived from acrylic acid. Such acrylic acid-based polymers which are useful herein are the water-soluble salts of polymerized acrylic acid. The average molecular weight of such polymers in the acid form preferably ranges from above 4,000 to 10,000, preferably from above 4,000 to 7,000, and most preferably from above 4,000 to 5,000. Water-soluble salts of such acrylic acid polymers can include, for example, the alkali metal, ammonium and substituted ammonium salts.

[0051] Co-polymeric polycarboxylates such as a Acrylic/maleic-based copolymers may also be used. Such materials include the water-soluble salts of copolymers of acrylic acid and maleic acid. The average molecular weight of such copolymers in the acid form preferably ranges from about 2,000 to 100,000, more preferably from about 5,000 to 75,000, most preferably from about 7,000 to 65,000. The ratio of acrylate to maleate segments in such copolymers will generally range from about 30:1 to about 1:1, more preferably from about 10:1 to 2:1. Water-soluble salts of such acrylic acid/maleic acid copolymers can include, for example, the alkali metal, ammonium and substituted ammonium salts. It is preferable for the above polymer solution to be pre-complexed with anionic surfactant such as LAS.

Adjunct Detergent Ingredients

[0052] The starting detergent material in the present process can include additional detergent ingredients and/or, any number of additional ingredients can be incorporated in the detergent composition during subsequent steps of the present process. These adjunct ingredients include other detergency builders, bleaches, bleach activators, suds boosters or suds suppressors, anti-tarnish and anticorrosion agents, soil suspending agents, soil release agents, germicides, pH adjusting agents, non-builder alkalinity sources, chelating agents, smectite clays, enzymes, enzyme-stabilizing agents and perfumes. See U.S. Patent 3,936,537, issued February 3, 1976 to Baskerville, Jr. et al., incorporated herein by reference.

[0053] Other builders can be generally selected from the various water-soluble, alkali metal, ammonium or substituted ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, borates, polyhydroxy sulfonates, polyacetates, carboxylates, and polycarboxylates. Preferred are the alkali metal, especially sodium, salts of the above. Preferred for use herein are the phosphates, carbonates, C_{10-18} fatty acids, polycarboxylates, and mixtures thereof. More preferred are sodium tripolyphosphate, tetrasodium pyrophosphate, citrate, tartrate mono- and di-succinates, and mixtures thereof (see below).

[0054] In comparison with amorphous sodium silicates, crystalline layered sodium silicates exhibit a clearly increased calcium and magnesium ion exchange capacity. In addition, the layered sodium silicates prefer magnesium ions over calcium ions, a feature necessary to insure that substantially all of the "hardness" is removed from the wash water. These crystalline layered sodium silicates, however, are generally more expensive than amorphous silicates as well as other builders. Accordingly, in order to provide an economically feasible laundry detergent, the proportion of crystalline layered sodium silicates used must be determined judiciously. Such crystalline layered sodium silicates are discussed in Corkill et al, U.S. Patent No. 4,605,509, previously incorporated herein by reference.

[0055] Specific examples of inorganic phosphate builders are sodium and potassium tripolyphosphate, pyrophosphate, polymeric metaphosphate having a degree of polymerization of from about 6 to 21, and orthophosphates. Examples of polyphosphonate builders are the sodium and potassium salts of ethylene diphosphonic acid, the sodium and potassium salts of ethane 1-hydroxy-1, 1-diphosphonic acid and the sodium and potassium salts of ethane, 1,1,2-triphosphonic acid. Other phosphorus builder compounds are disclosed in U.S. Patents 3,159,581; 3,213,030; 3,422,021; 3,422,137; 3,400,176 and 3,400,148, all of which are incorporated herein by reference.

[0056] Examples of nonphosphorus, inorganic builders are tetraborate decahydrate and silicates having a weight ratio of SiO_2 to alkali metal oxide of from about 0.5 to about 4.0, preferably from about 1.0 to about 2.4. Water-soluble, nonphosphorus organic builders useful herein include the various alkali metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates and polyhydroxy sulfonates. Examples of polyacetate and polycarboxylate builders are the sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylene diamine tetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, and citric add.

[0057] Polymeric polycarboxylate builders are set forth in U.S. Patent 3,308,067, Diehl, issued March 7, 1967, the disclosure of which is incorporated herein by reference. Such materials include the water-soluble salts of homo- and copolymers of aliphatic carboxylic acids such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, aconitic acid, citraconic acid and methylene malonic acid. Some of these materials are useful as the water-soluble anionic polymer as hereinafter described, but only if in intimate admixture with the non-soap anionic surfactant.

[0058] Other suitable polycarboxylates for use herein are the polyacetal carboxylates described in U.S. Patent 4,144,226, issued March 13, 1979 to Crutchfield et al, and U.S. Patent 4,246,495, issued March 27, 1979 to Crutchfield et al, both of which are incorporated herein by reference. These polyacetal carboxylates can be prepared by bringing together under polymerization conditions an ester of glyoxylic acid and a polymerization initiator. The resulting polyacetal carboxylate ester is then attached to chemically stable end groups to stabilize the polyacetal carboxylate against rapid depolymerization in alkaline solution, converted to the corresponding salt, and added to a detergent composition. Particularly preferred polycarboxylate builders are the ether carboxylate builder compositions comprising a combination of tartrate monosuccinate and tartrate disuccinate described in U.S. Patent 4,663,071, Bush et al., issued May 5, 1987, the disclosure of which is incorporated herein by reference.

[0059] Bleaching agents and activators are described in U.S. Patent 4,412,934, Chung et al., issued November 1, 1983, and in U.S. Patent 4,483,781, Hartman, issued November 20, 1984, both of which are incorporated herein by reference. Chelating agents are also described in U.S. Patent 4,663,071, Bush et al., from Column 17, line 54 through Column 18, line 68, incorporated herein by reference. Suds modifiers are also optional ingredients and are described in U.S. Patents 3,933,672, issued January 20, 1976 to Bartoletta et al., and 4,136,045, issued January 23, 1979 to Gault et al., both incorporated herein by reference.

[0060] Suitable smectite clays for use herein are described in U.S. Patent 4,762,645, Tucker et al, issued August 9, 1988, Column 6, line 3 through Column 7, line 24, incorporated herein by reference. Suitable additional detergency builders for use herein are enumerated in the Baskerville patent, Column 13, line 54 through Column 16, line 16, and in U.S. Patent 4,663,071, Bush et al, issued May 5, 1987, both incorporated herein by reference.

Optional Process Steps

[0061] Optionally, the process can comprise the step of spraying an additional binder in the mixer for the present invention. A binder is added for purposes of enhancing agglomeration by providing a "binding" or "sticking" agent for the detergent components. The binder is preferably selected from the group consisting of water, anionic surfactants, nonionic surfactants, liquid silicates, polyethylene glycol, polyvinyl pyrrolidone polyacrylates, citric acid and mixtures thereof. Other suitable binder materials including those listed herein are described in Beerse et al, U.S. Patent No. 5,108,646 (Procter & Gamble Co.), the disclosure of which is incorporated herein by reference.

[0062] Other optional steps contemplated by the present process include screening the oversized detergent agglomerates in a screening apparatus which can take a variety of forms including but not limited to conventional screens chosen for the desired particle size of the finished detergent product. Other optional steps include conditioning of the detergent agglomerates by subjecting the agglomerates to additional drying by way of apparatus discussed previously.

[0063] Another optional step of the instant process entails finishing the resulting detergent agglomerates by a variety of processes including spraying and/or admixing other conventional detergent ingredients. For example, the finishing step encompasses spraying perfumes, brighteners and enzymes onto the finished agglomerates to provide a more complete detergent composition. Such techniques and ingredients are well known in the art.

[0064] Another optional step in the process involves surfactant paste structuring process, e.g., hardening an aqueous anionic surfactant paste by incorporating a paste-hardening material by using an extruder, prior to the process of the present invention.

[0065] In order to make the present invention more readily understood, reference is made to the following examples, which are intended to be illustrative only and not intended to be limiting in scope.

EXAMPLES

Example 1:

[0066] The following is an example for obtaining agglomerates having high density, using Lödige CB mixer (CB-30), followed by Fluid Bed Apparatus for further granulations.

[0067] [Step 1] 250 - 270 kg/hr of aqueous coconut fatty alcohol sulfate surfactant paste (C_{12}-C_{18}, 71.5% active) is dispersed by the pin tools of a CB-30 mixer along with 220 kg/hr of powdered STPP (mean particle size of 40 - 75 microns), 160 - 200 kg/hr of ground soda ash (mean particle size of 15 microns). 80- 120 kg/hr of ground sodium sulfate (mean particle size of 15 microns), and the 200 kg/hr of internal recycle stream of powder. The surfactant paste is fed at about 40 to 52°C, and the powders are fed at room temperature. The condition of the CB-30 mixer is as follows:

Mean residence time : 10-18 seconds
Tip speed : 7.5 - 14 m/s
Energy condition : 0.5 - 4 kj/kg
Mixer speed : 550 - 900 rpm
Jacket temperature : 30°C

[0068] [Step 2] The agglomerates from the CB mixer are fed to a fluid bed drying apparatus for drying, rounding and growth of agglomerates. 20 - 120 kg/hr of liquid silicate (43% solids, 2.0R) is added in the fluid bed drying apparatus at 35°C. The condition of the fluid bed drying apparatus is as follows:

Mean residence time : 2 - 4 minutes
Depth of unfluidized bed : 200 mm
Droplet spray size : less than 50 micron
Spray height: 175 - 250 mm (above distributor plate)
Fluidizing velocity : 0.4 - 0.8 m/s
Bed temperature : 40 - 70 °C]

The resulting granules from the step 2 have a density of about 600 g/l, and can be optionally subjected to the optional process of cooling, sizing and/or grinding.

Example 2:

[0069] The following is an example for obtaining agglomerates having high density, using Lödige CB mixer (CB-30), followed by Fluid Bed Apparatus for further granulaions.
[0070] [Step 1] 15 kg/hr - 30kg/hr of HLAS (an acid precursor of C_{11}-C_{18} alkyl benzene sulfonate; 95% active) at about 50 °C, and 20 kg/hr of AE_3S liquid (C_{10}-C_{18} alkyl alkoxy sulfates, EO-3; 28% active) is dispersed by the pin tools of a CB-30 mixer along with 220 kg/hr of powdered STPP (mean particle size of 40 - 75 microns), 160 - 200 kg/hr of ground soda ash (mean particle size of 15 microns), 80- 120 kg/hr of ground sodium sulfate (mean particle size of 15 microns), and the 200 kg/hr of internal recycle stream of powder. The surfactant paste is fed at about 40 to 52°C, and the powders are fed at room temperature.
The condition of the CB-30 mixer is as follows:

Mean residence time : 10-18 seconds
Tip speed : 7.5 - 14 m/s
Energy condition : 0.5 - 4 kj/kg
Mixer speed : 550 - 900 rpm
Jacket temperature : 30°C

[0071] [Step 2] The agglomerates from the CB-30 mixer are fed to a fluid bed drying apparatus for drying, rounding and growth of agglomerates. 20 - 80 kg/hr of liquid silicate (43% solids, 2.0 R) is added in the fluid bed drying apparatus at 35°C. The condition of the fluid bed drying apparatus is as follows:

Mean residence time : 2- 4 minutes
Depth of unfluidized bed : 200 mm
Droplet spray size : less than 50 micron
Spray height 175 - 250 mm (above distributor plate)
Fluidizing velocity : 0.4 -.0.8 m/s
Bed temperature : 40 - 70 °C

The resulting granules from the step 2 have a density of about 600 g/l, and can be optionally subjected to the optional processes of cooling, sizing an/or grinding.

Example 3:

[0072] The following is an example for obtaining agglomerates having high density, using Lödige CB mixer (CB-30), followed by Fluid Bed Apparatus for further granulations.
[0073] [Step 1] 15 kg/hr - 30kg/hr of HLAS (an acid precursor of C_{11}-C_{18} alkyl benzene sulfonate; 95% active) at about 50 °C, and 250 - 270 kg/hr of aqueous coconut fatty alcohol sulfate surfactant paste (C_{12}-C_{18}, 71.5% active) is

dispersed by the pin tools of a CB-30 mixer along with 220 kg/hr of powdered STPP (mean particle size of 40 - 75 microns), 160 - 200 kg/hr of ground soda ash (mean particle size of 15 microns), 80-120 kg/hr of ground sodium sulfate (mean particle size of 15 microns), and the 200 kg/hr of internal recycle stream of powder. The surfactant paste is fed at about 40 to 52°C, and the powders are fed at room temperature. The condition of the CB-30 mixer is as follows:

Mean residence time : 10 - 18 seconds
Tip speed : 7.5 - 14 m/s
Energy condition : 0.5 - 4 kj/kg
Mixer speed : 550 - 900 rpm
Jacket temperature : 30°C

[0074] [Step 2] The agglomerates from the CB-30 mixer are fed to a fluid bed drying apparatus for drying, rounding and growth of agglomerates. 20 - 80 kg/hr of liquid silicate (43% solids, 2.0 R) is added in the fluid bed drying apparatus at 35°C. The condition of the fluid bed drying apparatus is as follows:

Mean residence time : 2- 4 minutes
Depth of unfluidized bed : 200 mm
Droplet spray size : less than 50 micron
Spray height 175 - 250 mm (above distributor plate)
Fluidizing velocity: 0.4 - 0.8 m/s
Bed temperature : 40 - 70 °C

[0075] The resultant from the fluid bed drying apparatus is fed to a fluid bed cooling apparatus. The condition of the fluid bed cooling apparatus is as follows:

Mean residence time : 2- 4 minutes
Depth of unfluidized bed : 200 mm
Fluidizing velocity : 0.4 - 0.8 m/s
Bed temperature : 12 - 60 °C]

The resulting granules from the step 2 has a density of about 600 g/l, and can be optionally subjected to the optional process of sizing an/or grinding.

Claims

1. An agglomeration process for preparing a granular detergent composition having a density of at least about 600 g/l, comprising the steps of:

   (a) dispersing a surfactant, and coating the surfactant with fine powder having a diameter from 0.1 to 500 microns, in a mixer wherein conditions of the mixer include (i) from 2 to 50 seconds of mean residence time, (ii) from 4 to 25 m/s of tip speed, and (iii) from 0.15 to 7 kj/kg of energy condition, wherein agglomerates are formed; and
   (b) granulating the agglomerates in fluidised bed drying apparatus while spraying into the bed droplets of liquid detergent material in an amount of up to 20%, wherein conditions of the fluidising apparatus include (i) from 1 to 10 minutes of mean residence time, (ii) from 100 to 300 mm of depth of unfluidised bed, (iii) not more than 50 micron of droplet spray size, (iv) from 175 to 250mm of spray height, (v) from 0.2 to 1.4 m/s of fluidising velocity and (vi) from 12 to 100°C of bed temperature.

2. A process according to claim 1 in which 2 to 10% (active basis) of liquid detergent material is sprayed into the bed.

3. A process according to claim 1 or claim 2 in which the sprayed liquid detergent material is selected from solutions of silicate, anionic surfactant, cationic surfactant, and polymer.

4. A process according to any preceding claim in which a coating agent, to improve flowability or to minimise over granulation, is added directly after the fluidised bed drying apparatus.

5. A process according to any of claims 1 to 3 in which the fluidised bed drying apparatus is followed by a fluidised

bed cooler.

6. A process according to claim 5 in which a coating agent, to improve flowability or to minimise over granulation, is added between the fluidised bed drying apparatus and the fluidised cooler or directly after the fluidised bed cooler.

7. A process according to claim 1 wherein an aqueous or non-aqueous polymer solution is dispersed with said surfactant in step (a).

8. A process according to claim 1 wherein an internal recycle stream of powder from the fluidising apparatus is further added to step (a).

9. A process according to calim 1 wherein the fine powder is selected from the group consisting of soda ash, powdered sodium tripolyphosphate, hydrated tripolyphosphate, sodium sulphates, aluminosilicates, crystalline layered silicates, phosphates, precipitated silicates, polymers, carbonates, citrates, nitrilotriacetates, powdered surfactants and mixtures thereof.

10. A process according to claim 1 wherein total amount of surfactants for the process of claim 1 is from about 5% to about 60%, in total amount of a composition obtained by the process of claim 1.

Patentansprüche

1. Agglomerationsverfahren zur Herstellung einer granulären Detergenszusammensetzung mit einer Dichte von mindestens etwa 600 g/l, umfassend die Schritte:

   (a) Dispergieren eines Tensids, und Beschichten des Tensids mit feinem Pulver mit einem Durchmesser von 0.1 bis 500 μm in einem Mischer, wobei Bedingungen des Mischers beinhalten (i) 2 bis 50 Sekunden mittlere Verweilzeit, (ii) 4 bis 25 m/s Spitzengeschwindigkeit und (iii) 0,15 bis 7 kj/kg Energiezustand, wobei Agglomerate gebildet werden; und
   (b) Granulieren der Agglomerate in einer Wirbelbett-Trocknungsvorrichtung, während Tropfen flüssigen Detergensmaterials in einer Menge von bis zu 20% in das Bett gesprüht werden, wobei Bedingungen der Wirbelvorrichtung umfassen (i) 1 bis 10 Minuten mittlere Verweilzeit, (ii) 100 bis 300 mm Tiefe an nichtfluidisiertem Bett, (iii) nicht mehr als 50 μm Tropfensprühgröße, (iv) 175 bis 250 mm Sprühhöhe, (v) 0,2 bis 1,4 m/s Wirbelgeschwindigkeit und (vi) 12 bis 100°C Betttemperatur.

2. Verfahren nach Anspruch 1, wobei 2 bis 10% (Wirkstoffbasis) flüssiges Detergensmaterial in das Bett gesprüht werden.

3. Verfahren nach Anspruch 1 oder Anspruch 2, wobei das gesprühte flüssige Detergensmaterial gewählt ist aus Lösungen von Silicat, anionisches Tensid, kationisches Tensid und Polymer.

4. Verfahren nach mindestens einem der vorangehenden Ansprüche, wobei ein Beschichtungsmittel direkt nach der Wirbelbett-Trocknungsvorrichtung zugegeben wird, um die Fließfähigkeit zu verbessern oder Übergranulation zu minimieren.

5. Verfahren nach mindestens einem der Ansprüche 1 bis 3, wobei die Wirbelbett-Trocknungsvorrichtung gefolgt wird von einem Wirbelbett-Kühler.

6. Verfahren nach Anspruch 5, wobei ein Beschichtungsmittel zwischen der Wirbelbett-Trocknungsvorrichtung und dem Wirbelbett-Kühler oder direkt nach dem Wirbelbett-Kühler zugesetzt wird, um Fließfähigkeit zu verbessern, oder Übergranulation zu minimieren.

7. Verfahren nach Anspruch 1, wobei eine wässrige oder nichtwässrige Polymerlösung mit dem Tensid in Schritt (a) dispergiert wird.

8. Verfahren nach Anspruch 1, wobei ein interner Kreislaufstrom aus Pulver aus der Wirbelbett-Vorrichtung weiterhin Schritt (a) zugesetzt wird.

9. Verfahren nach Anspruch 1, wobei das feine Pulver aus der Gruppe gewählt wird, bestehend aus Sodaasche, pulverförmiges Natriumtripolyphosphat, hydratisiertes Tripolyphosphat, Natriumsulfaten, Aluminosilicaten, kristallinen Schichtsilicaten, Phosphaten, ausgefällten Silicaten, Polymeren, Carbonaten, Citraten, Nitrilotriacetaten, pulverförmigen Tensiden und Mischungen hiervon.

10. Verfahren nach Anspruch 1, wobei die Gesamtmenge an Tensiden für das Verfahren nach Anspruch 1 etwa 5% bis etwa 60% der Gesamtmenge einer Zusammensetzung, wie durch das Verfahren nach Anspruch 1 erhalten, beträgt.

Revendications

1. Procédé d'agglomération pour préparer une composition détergente granulaire ayant une masse volumique d'au moins environ 600 g/l, comprenant les étapes consistant :

   (a) à disperser un agent tensioactif, et à revêtir l'agent tensioactif avec une poudre fine ayant un diamètre de 0,1 à 500 microns, dans un mélangeur dans lequel les conditions de mélange incluent (i) de 2 à 50 secondes de temps de séjour moyen, (ii) de 4 à 25 m/s de vitesse en extrémité, et (iii) de 0,15 à 7 kj/kg de condition énergétique, dans laquelle des agglomérats sont formés ; et

   (b) à granuler les agglomérats dans un appareil de séchage à lit fluidisé tout en pulvérisant dans le lit des gouttelettes de matière détergente liquide en une quantité jusqu'à 20%, dans laquelle les conditions de l'appareil de fluidisation incluent (i) de 1 à 10 minutes de temps de séjour moyen, (ii) de 100 à 300 mm de profondeur de lit non fluidisé, (iii) pas plus de 50 microns de taille de gouttelette pulvérisée, (iv) de 175 à 250 mm de hauteur de pulvérisation, (v) de 0,2 à 1,4 m/s de vitesse de fluidisation et (vi) de 12 à 100 °C de température de lit.

2. Procédé selon la revendication 1, dans lequel 2 à 10 % (sur une base active) de matière détergente liquide est pulvérisée dans le lit.

3. Procédé selon la revendication 1 ou la revendication 2, dans lequel la matière détergente liquide pulvérisée est choisie parmi les solutions de silicate, d'agent tensioactif anionique, d'agent tensioactif cationique et de polymère.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel un agent de revêtement, pour améliorer l'aptitude à l'écoulement ou minimiser une sur-granulation, est ajouté directement après l'appareil de séchage à lit fluidisé.

5. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel l'appareil de séchage à lit fluidisé est suivi par un refroidisseur à lit fluidisé.

6. Procédé selon la revendication 5, dans lequel un agent de revêtement, pour améliorer l'aptitude à l'écoulement ou pour minimiser une sur-granulation, est ajouté entre l'appareil de séchage à lit fluidisé et le refroidisseur à lit fluidisé ou directement après le refroidisseur à lit fluidisé.

7. Procédé selon la revendication 1, dans lequel une solution polymère aqueuse ou non aqueuse est dispersée avec ledit agent tensioactif dans l'étape (a).

8. Procédé selon la revendication 1, dans lequel un courant de recyclage interne de poudre à partir de l'appareil de fluidisation est en outre ajouté à l'étape (a).

9. Procédé selon la revendication 1, dans lequel la poudre fine est choisie dans le groupe constitué par le carbonate de sodium, le tripolyphosphate de sodium pulvérulent, le tripolyphosphate hydraté, les sulfates de sodium, les aluminosilicates, les silicates stratifiés cristallins, les phosphates, les silicates précipités, les polymères, les carbonates, les citrates, les nitrilotriacétates, les agents tensioactifs pulvérulents et des mélanges de ceux-ci.

10. Procédé selon la revendication 1, dans lequel la quantité totale d'agents tensioactifs pour le procédé de la revendication 1 est d'environ 5 % à environ 60 %, en quantité totale d'une composition obtenue par le procédé de la revendication 1.