Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).
BACKGROUND 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.

[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" 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" 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
arating the agglomerates according to particle size and either grinding or recycling agglomerates for further agglom-
eration 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.

None of the existing art provides all of the advantages and benefits of the present invention.

SUMMARY OF THE INVENTION

The present invention meets the aforementioned needs in the art by providing a process which produces a high density granular detergent 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.

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 detergent ingredients in the form of liquid, into the process.

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.


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. Composition. The present invention also meets the aforementioned needs in the art by providing a process which produces a granular detergent composition for flexibility in the ultimate density of the final composition from agglomeration (e.g., non-tower) process. 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.

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

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

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".

In accordance with one aspect of the invention, a process for preparing a granular detergent composition having a density at least about 600 g/l is provided. 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, while wetting the surfactant coated with the fine powder with finely atomized liquid, in a mixer wherein conditions of the mixer include (i) from 0.2 to 5 seconds of mean residence time, (ii) from 15 to 26 m/s of tip speed, and (iii) from 0.2 to 3 kJ/kg of energy condition, wherein agglomerates are formed; and (b) granulating the agglomerates in a fluid bed dryer and fluid bed cooler wherein conditions of each of the fluidizing apparatus include (i) from 1 to 10 minutes of mean residence time, (ii) from 100 to 300 mm of depth of unfluidized
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 fluidizing velocity and (vi) from 12 to 100 °C of bed temperature, wherein the mean residency time of step (b) in total is from 2 to 12 minutes, and wherein a coating agent selected from the group consisting of aluminosilicates, silicates, carbonates and mixtures thereof is added to step (b) either: directly after the fluid bed cooler or fluid bed dryer; and/or between the fluid bed dryer and fluid bed cooler; and/or directly to the fluid bed dryer.

[0018] Also provided are the granular detergent compositions having a high density of at least 600g/l, produced by any one of the process embodiments described herein.

[0019] Accordingly, it is an object of the invention to provide a process for continuously producing a detergent composition which has flexibility with respect to density of the final products by controlling energy input, residence time condition, and tip speed condition in the mixers. It is also an object of the invention to provide a process which is more efficient, flexible and economical to facilitate large-scale production. These and other objects, features and attendant advantages of the present invention will become apparent to those skilled in the art from a reading of the following detailed description of the preferred embodiment and the appended claims.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[0020] The present invention is directed to a process which produces free flowing, granular detergent agglomerates having a density of at least 600 g/l. The process produces granular detergent agglomerates from an aqueous and/or non-aqueous surfactant which is then coated with fine powder having a diameter from 0.1 to 500 microns, in order to obtain low density granules.

Process

First Step [Step(a)]

[0021] In the first step of the process, 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 1 to 100 microns are fed into a mixer, so as to make agglomerates. During the process, surface of the surfactant which is coated by the fine powder is wet by finely atomized liquid so as to add more fine powder on the surface of the agglomerates. (The definition of the surfactants and the fine powder, finely atomized liquid are described in detail hereinafter.) Optionally, an internal recycle stream of powder having a diameter of 0.1 to 300 microns generated in the fluidizing apparatus (e.g., fluid bed dryer and/or fluid bed cooler) 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 60 wt% of final product.

[0022] In another embodiment of the invention, the surfactant for the first step can be initially fed into a mixer or pre-mixer (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.

[0023] Generally speaking, preferably, the mean residence time of the mixer is in range from 0.2 to 5 seconds and tip speed of the mixer of the mixer is in range from 10 m/s to 30 m/s, the energy per unit mass of the mixer (energy condition) of the mixer is in range from 0.15 kJ/kg to 5 kJ/kg, more preferably, the mean residence time of the mixer is in range from 0.2 to 5 seconds and tip speed of the mixer is in range from 10 m/s to 30 m/s, the energy per unit mass of the mixer (energy condition) is in range from 0.15 kJ/kg to 5 kJ/kg, the most preferably, the mean residence time of the mixer is in range from 0.2 to 5 seconds, tip speed of the mixer is in range from 15 m/s to 26 m/s, the energy per unit mass of the mixer (energy condition) is from 0.2 kJ/kg to 3 kJ/kg.

[0024] The examples of the mixer 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 Flexomic Model manufactured by the Schugi company (Netherlands).

Second Step [Step(b)]

[0025] In the second step of the process, the agglomerates from the first step are fed into a fluidized apparatus, such as fluidized bed, in order to enhance granulation for producing free flowing high density granules. The second step can proceed in one or more than one fluidized apparatus (e.g., combining different kinds of fluidized apparatus such as fluid bed dryer and fluid bed cooler). In the second step, the resultant product from the second step is fluidized thoroughly so that the granules from the second step have a round shape. Optionally, 0 to 10%, more preferably 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. Also, optionally, 0 to 20%, more preferably 2 to 10% of liquid detergent materials of the kind used in the first step, the second step and/or other detergent ingredients can be added to the step, for enhancing granulation and
coating on the surface of the granules.

[0026] Generally speaking, to achieve the density of at least 600 g/l, preferably more than 650g/l, condition of a fluidized apparatus can be;

Mean residence time: from 1 to 10 minutes
Depth of unfluidized bed: from 100 to 300 mm
Droplet spray size: not more than 50 micron
Spray height: from 175 to 250 mm
Fluidizing velocity: from 0.2 to 1.4 m/s
Bed temperature: from 12 to 100 °C,

more preferably;

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

[0027] If two different kinds of fluidized apparatus would be used, mean residence time of the third step in total can be from 2 to 20 minutes, more preferably, from 2 to 12 minutes.

[0028] A coating agent to improve flowability and/or minimize over agglomeration of the detergent composition is added in one or more of the following locations of the instant process: (1) the coating agent can be added directly after fluid bed cooler or fluid bed dryer; (2) the coating agent may be added between fluid bed dryer and fluid bed cooler; and/or (3) the coating agent may be added directly to the fluid bed dryer. 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

[0029] 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 5% to 60%, more preferably from 12% to 40%, more preferably, from 15 to 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 (Aqueous /Non-aqueous)

[0030] The amount of the surfactant of the present process can be from 5% to 60%, more preferably from 12% to 40%, more preferably, from 15 to 35%, in total amount of the final product obtained by the process of the present invention.

[0031] 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.

[0032] 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.

[0033] Nonlimiting examples of the preferred anionic surfactants useful in the present invention include the conventional C_{11-18} alkyl benzene sulfonates ("LAS"), primary, branched-chain and random C_{10-12} alkyl sulfates ("AS"), the C_{10-12} secondary (2,3) alkyl sulfates of the formula \( \text{CH}_\text{x}(\text{CH}_\text{2})\text{y}(\text{CHOSO}_3\text{-M}^+) \text{CH}_\text{2} \text{CH}_\text{2} \text{CH}_\text{2} \) where x and (y + 1) are integers of at least 7, preferably at least 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, and the C_{10-12} alkyl alkoxy sulfates ("AE\text{S}"), especially
Useful anionic surfactants also include water-soluble salts of 2-acyloxy-alkane-1-sulfonic acids containing from 2 to 9 carbon atoms in the acyl group and from 9 to 23 carbon atoms in the alkane moiety; water-soluble salts of olefin sulfonates containing from 12 to 24 carbon atoms; and beta-alkyloxy alkane sulfonates containing from 1 to 3 carbon atoms in the alkyl group and from 8 to 20 carbon atoms in the alkane moiety.

Optionally, other exemplary surfactants useful in the paste of the invention include C_{10}-C_{18} alkyl alkoxy carboxylates (especially the EO 1-5 ethoxy carboxylates; the C_{18}-C_{16} glycerol ethers, the C_{10}-C_{16} 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 ethoxylates (especially ethoxylates and mixed ethoxy/propoxy), C_{10}-C_{18} amine oxides, 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-methyl glucamides. 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_{18} 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.

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. 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')_{2}N^+R_{2}SO_{3}^{-}, wherein R is a C_{6}-C_{18} hydrocarbyl group, preferably a C_{10}-C_{16} alkyl group or C_{18}-C_{16} amido alkyl group, each R' 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} alkenyl group, more preferably a C_{1}-C_{2} alkenyl group. Examples of suitable betaines include coconut acylamidopropyl dimethyl betaine; hexadecyl dimethyl betaine; C_{12-14} acylamidopropyl betaine; C_{6-18} acylamidoheptyldiethyldiethyldiethyl betaine; [C_{4-14} acylamidopropyl]betaine. Preferred betaines are C_{12-18} dimethyl-ammonia hexaenoate and the C_{10-18} acylamidopropene (or ethene) dimethyl (or ethylenic) betaines; and the suftaines having the formula (R(R')_{2}N^+R_{2}SO_{3}^{-}, wherein R is a C_{6}-C_{18} hydrocarbyl group, preferably a C_{10}-C_{16} alkyl group, preferably a C_{12}-C_{14} alkyl group, each R' is typically C_{1}-C_{3} alkyl, preferably methyl, and R_{2} is a C_{1}-C_{8} hydrocarbyl group, preferably a C_{1}-C_{3} alkenyl or, preferably, hydroxyalkylene group. Examples of suitable suftaines include C_{12}-C_{14} dimethylammonio-2-hydroxypropyl sulfonate, C_{12}-C_{14} amido propyl ammonio-2-hydroxypropyl sultaine, C_{12}-C_{14} dimethyldiammonio propene sulfonate, and C_{16}-C_{18} dimethyldiammonio hexane sulfonate, with C_{12-14} amido propyl ammonio-2-hydroxypropyl sultaine being preferred.

The amount of the fine powder of the present process, which is used in the first step, can be from 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), the disclosure of which is incorporated herein by reference.

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 prefer-
ably 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 mi-
croscope (SEM). The preferred particle size diameter of the aluminosilicate is from 0.1 micron to 10 microns, more
preferably from 0.5 microns to 9 microns. Most preferably, the particle size diameter is from 1 microns to 8 microns.

[0039] Preferably, the aluminosilicate ion exchange material has the formula Na\textsubscript{z}[(AlO\textsubscript{2})\textsubscript{y}·(SiO\textsubscript{2})\textsubscript{x}]H\textsubscript{2}O
wherein z and y are integers of at least 6, the molar ratio of z to y is from 1 to 5 and x is from 10 to 264. More preferably,
the aluminosilicate has the formula

\[ \text{Na}_{12}[(\text{AlO}_2)_{12}·(\text{SiO}_2)_{12}]\times\text{H}_2\text{O} \]

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

[0040] The aluminosilicates used herein are further characterized by their ion exchange capacity which is at least
200 mg equivalent of CaCO\textsubscript{3} hardness/gram, calculated on an anhydrous basis, and which is preferably in a range
from 300 to 352 mg equivalent of CaCO\textsubscript{3} hardness/gram.

Finely Atomized Liquid

[0041] The amount of the finely atomized liquid of the present process can be from 1% to 10% (active basis), pref-
erably from 2% to 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, water and mixtures thereof.
Other optional examples for the finely atomized liquid of the present invention can be sodium cocoxy methyl cellulose
solution, polyethylene glycol (PEG), and solutions of dimethylene triamine pentamethyl phosphonic acid (DETMP).

[0042] The preferable examples of the anionic surfactant solutions which can be used as the finely atomized liquid
in the present inventions are 88 - 97% active HLAS, 30 - 50% active NaLAS, 28% active AE3S solution, 40-50% active
liquid silicate.

[0043] Cationic surfactants can also be used as finely atomized liquid herein and suitable quaternary ammonium
surfactants are selected from mono C\textsubscript{6}-C\textsubscript{18}, preferably C\textsubscript{6}-C\textsubscript{10} N-alkyl or alkanyl ammonium surfactants wherein re-
maining N positions are substituted by methyl, hydroxyethyl or hydroxypropyl groups.

[0044] Preferable examples of the aqueous or non-aqueous polymer solutions which can be used as the finely at-
omized liquid in the present inventions are modified polyamines which comprise a polyamine backbone corresponding
to the formula:

\[
\text{H-N-R}_m\text{N}^+\text{R}_m\text{N}^+\text{R}_m\text{N}_m\text{NH}_2
\]

having a modified polyamine formula V\textsubscript{(n+1)}W\textsubscript{m}Y\textsubscript{n}Z or a polyamine backbone corresponding to the formula:

\[
\text{H-N-R}_m\text{N}^+\text{R}_m\text{N}^+\text{R}_m\text{N}_m\text{K}^-\text{NH}_2
\]

having a modified polyamine formula V\textsubscript{(n+k+1)}W\textsubscript{m}Y\textsubscript{n}Y'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:

\[
\begin{align*}
E &-N-R^- \quad \text{or} \quad E &-N-R^- \quad \text{or} \quad E &-N-R^- \\
\end{align*}
\]

iii) Y units are branching units having the formula:

\[
\begin{align*}
N &-R^- \quad \text{or} \quad N &-R^- \quad \text{or} \quad N &-R^- \\
\end{align*}
\]

and

iv) Z units are terminal units having the formula:

\[
\begin{align*}
-N^- \quad \text{or} \quad N^+ \quad \text{or} \quad N^+ \\
\end{align*}
\]

wherein backbone linking R units are selected from the group consisting of C₂₋₁₂ alkyne, C₂₋₁₂ alkenylene, C₂₋₁₂ hydroxalkylene, C₂₋₁₂ dihydroxyalkylene, C₂₋₁₂ dialkylarylene, -(R¹O)ₓR¹⁻, -(R¹O)ₓR⁵(OR¹)x⁻, -(CH₂CH(OR²)CH₃)ₓ(OR¹)ₓ(OR⁵)ₓ(OR²)ₓCH₃⁻, -(CH₂CH(OR²)CH₃)ₓ(OR¹)ₓ(OR⁵)ₓ(OR²)ₓCH₃⁻, and mixtures thereof; wherein R¹ is C₂₋₆ alkyne and mixtures thereof; R² is hydrogen, -(R¹O)ₓB, and mixtures thereof; R³ is C₁₋₁₆ alkyl, C₇₋₁₆ alkyllhal, C₇₋₁₂ alkyl substituted aryl, C₆₋₁₂ aryl, and mixtures thereof; R⁴ is C₁₋₁₂ alkyne, C₆₋₁₂ hydroxyalkylene, C₆₋₁₂ dihydroxyalkylene, C₆₋₁₂ dialkylarylene, -(R¹O)ₓR¹⁻, -(R¹O)ₓR³(OR¹)x⁻, -(CH₂CH(OR²)CH₃)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, -(CH₂CH(OR²)CH₃)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, and mixtures thereof; R⁵ is C₂₋₁₂ alkyl, C₂₋₁₂ arylen, C₂₋₁₂ hydroxalkylene, C₂₋₁₂ dihydroxalkylene, C₂₋₁₂ dialkylarylene, -(O⁻), -(OR¹)ₓN¹HₓNHC(OR₁)(OR₁)ₓ⁻, -(OR¹)ₓ(OR¹)ₓ⁻, -(OR¹)ₓR¹⁻, -(OR¹)ₓR³⁻, -(CH₂CH(OR²)CH₃)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, -(CH₂CH(OR²)CH₃)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, and mixtures thereof; R⁶ is C₂₋₁₂ alkyne or C₂₋₁₂ arylen; E units are selected from the group consisting of hydrogen, C₁₋₂₂ alkyne, C₁₋₂₂ aryl, C₁₋₂₂ alkyllhal, C₂₋₂₂ hydroxalkyl, -(CH₂)ₓCO₂M⁻, -(CH₂)ₓSO₂M⁻, -(CH₂)ₓCO₂M⁻, -(CH₂)ₓPO₄M⁻, -(R¹O)ₓB, -(OR¹)ₓ⁻, -(OR¹)ₓ⁻, -(CH₂)ₓ(OR²)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, -(CH₂)ₓ(OR²)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, and mixtures thereof; M is hydrogen, -(CH₂)ₓ(OR²)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, -(CH₂)ₓ(OR²)ₓ(OR¹)ₓ(OR³)ₓ(OR²)ₓCH₃⁻, 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 400; n has the value from 0 to 200; p has the value from 1 to 6; q has the value from 0 to 6; r has the value from 0 to 1; w has the value from 1 to 100; y has the value from 0 to 100; y has the value from 0 to 1; one example of the most preferred polyethylenimines 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.

[0045] 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 polym-
erizing 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 40% by weight of the polymer.

[0046] 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.

[0047] 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 2,000 to 100,000, more preferably from 5,000 to 75,000, most preferably from 7,000 to 65,000. The ratio of acrylate to maleate segments in such copolymers will generally range from 30:1 to 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

[0048] 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, antitarnish 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.

[0049] Other builders can be generally selected from the various water-soluble, alkaline metal, ammonium or substituted ammonium phosphates, phosphonates, polyphosphonates, carbonates, borates, polyhydroxy sulfonates, polycarboxylates, 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).

[0050] 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.

[0051] Specific examples of inorganic phosphate builders are sodium and potassium tripolyphosphate, pyrophosphate, polymeric metaphosphate having a degree of polymerization of from 6 to 21, and orthophosphates. Examples of polyphosphonate builders are the sodium and potassium salts of ethylene di-phosphonic acid, the sodium and potassium salts of ethane 1,1-hydroxy-1,1-diphosphonic acid and the sodium and potassium salts of ethylene, 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.

[0052] Examples of nonphosphorus, inorganic builders are tetraborate decahydrate and silicates having a weight ratio of SiO_{2} to alkali metal oxide of from 0.5 to 4.0, preferably from 1.0 to 2.4. Water-soluble, nonphosphorus organic builders useful herein include the various alkali metal, ammonium and substituted ammonium polycarboxylates, carboxylates, polycarboxylates and polyhydroxy sulfonates. Examples of polycarboxylate and polycarboxylate builders are the sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylene diamine tetraacetic acid, nitritrolactic acid, oxdysuccinic acid, melletic acid, benzene polycarboxylic acids, and citric acid.

[0053] Polymeric polycarboxylate builders are set forth in U.S. Patent 3,308,067, Diehl, issued March 7, 1967. Such materials include the water-soluble salts of homo- and copolymers of aliphatic carboxylic acids such as maleic acid, itaconic acid, fumaric acid, maleic anhydride, citraconic acid and methylenemalonic 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.
Other suitable polycarboxylates for use herein are the polycetal 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. These polycetal carboxylates can be prepared by bringing together under polymerization condition an ester of glyoxylic acid and a polymerization initiator. The resulting polycetal carboxylate ester is then attached to chemically stable end groups to stabilize the polycetal 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.

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. 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. 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..

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. 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.

Optional Process Steps

Optionally, the process can comprise the step of spraying an additional binder in one or more than one of the first, second and/or the third mixers 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 polycrylates, citric acid and mixtures thereof. Other suitable binder materials including those listed herein are described in Beese et al, U.S. Patent No. 5,108,646 (Procter & Gamble Co.).

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.

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.

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.

EXAMPLES

Example 1:

The following is an example for obtaining agglomerates having high density, using Schugi FX-160 Mixer, followed by Fluid Bed Apparatus for further granulations.

[Step 1] 120 - 160 kg/hr of HLAS (an acid precursor of C11-C18 alkyl benzene sulfonate; 96% active) is dispersed in a highly turbulent air stream of the Schugi FX-160 mixer along with 220 kg/hr of powdered STPP (mean particle size of 40 - 75 microns), 160 - 280 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 is fed at about 50 to 60 °C, and the powders are fed at room temperature. Then, 30 kg/hr of HLAS (an acid precursor of C11-C18 alkyl benzene sulfonate; 94 - 97% active) is dispersed as finely atomized liquid in the FX-160 mixer at about 50 to 60°C. 20-80 kg/hr of soda ash (mean particle size of about 10 - 20 microns) is added in the Schugi mixer. The condition of the Schugi mixer is as follows:

- Mean residence time : 0.2 - 5 seconds
- Tip speed : 16 - 26 m/s
- Energy condition : 0.15 - 2 kj/kg
- Mixer speed : 2000 - 3200 rpm

The surfactant paste is structuring process is as follows:

Mean residence time : 0.2 - 5 seconds
Tip speed : 16 - 26 m/s
Energy condition : 0.15 - 2 kj/kg
Mixer speed : 2000 - 3200 rpm
Step 2: The agglomerates from the Schugi 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) can be also 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 resultant from the step 2 has a density of about 600g/l, and can be optionally subjected to the optional process of cooling, sizing and/or grinding.

Example 2:

0062 The following is an example for obtaining agglomerates having high density, using Schugi FX-160 Mixer, followed by Fluid Bed Apparatus for further agglomerations.

Step 1: 120 - 160 kg/hr of HLAS (an acid precursor of C_{11}-C_{18} alkyl benzene sulfonate; 95% active) at about 50°C, is dispersed in a highly turbulent air stream of the Schugi FX-160 mixer along with 220 kg/hr of powdered STPP (mean particle size of 40 - 75 microns), 160 - 280 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 condition of the Schugi mixer is as follows:

- Mean residence time: 0.2 - 5 seconds
- Tip speed: 16 - 26 m/s
- Energy condition: 0.15 - 2 kj/kg
- Mixer speed: 2000 - 3200 rpm

Step 2: The agglomerates from the Schugi 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) can be also 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

Step 3: The resultant from the fluid bed drying apparatus is fed to a fluid bed cooling apparatus. 5 - 10 kg/hr of liquid silicate (43% solids, 2.0 R) is added to the 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 resultant from the step 3 has a density of about 600g/l, and can be optionally subjected to the optional process of sizing and/or grinding.

0063 Having thus described the invention in detail, it will be obvious to those skilled in the art that various changes may be made without departing from the scope of the invention and the invention is not to be considered limited to what is described in the specification.
Claims

1. A non-tower process for preparing a granular detergent composition having a density of at least 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, while wetting the surfactant coated with the fine powder with finely atomized liquid, in a mixer wherein conditions of the mixer include (i) from 0.2 to 5 seconds of mean residence time, (ii) from 15 to 26 m/s of tip speed, and (iii) from 0.2 to 3 kJ/kg of energy condition, wherein agglomerates are formed; and
   (b) granulating the agglomerates in a fluid bed dryer and fluid bed cooler

   wherein conditions of each of the fluidizing apparatus include (i) from 1 to 10 minutes of mean residence time, (ii) from 100 to 300 mm of depth of unfluidized 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 fluidizing velocity and (vi) from 12 to 100 °C of bed temperature, wherein the mean residency time of step (b) in total is from 2 to 12 minutes, and wherein a coating agent selected from the group consisting of aluminosilicates, silicates, carbonates and mixtures thereof is added to step (b) either: directly after the fluid bed cooler or fluid bed dryer; and/or between the fluid bed dryer and fluid bed cooler; and/or directly to the fluid bed dryer.

2. The process according to claim 1 wherein said surfactant is selected from the group consisting of anionic surfactant, nonionic surfactant, cationic surfactant, zwitterionic, ampholytic and mixtures thereof.

3. The process according to claim 1 wherein said surfactant is selected from the group consisting of alkyl benzene sulfonates, alkyl alkoxy sulfates, alkyl ethoxylates, alkyl sulfates, coconut fatty alcohol sulfates and mixtures thereof.

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

5. The process according to claim 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.

6. The process according to claim 1 wherein the finely atomized liquid is selected from the group consisting of liquid silicates, anionic surfactants, cationic surfactants, aqueous polymer solutions, non-aqueous polymer solutions, water and mixtures thereof.

7. The process according to claim 1 wherein an internal recycle stream of powder from the fluidizing apparatus is further added to step (a).

Patentansprüche

1. Nicht-Turm-Verfahren zur Herstellung einer granulären Detergenszusammensetzung mit einer Dichte von mindestens 600 g/l, umfassend die Schritte:

   (a) Dispergieren eines Tensids und Beschichten des Tensids mit feinem Pulver eines Durchmessers von 0,1 bis 500 µm, während das mit feinem Pulver beschichtete Tensid mit feinzerstäubter Flüssigkeit benetzt wird, in einem Mischer, wobei Bedingungen des Mischers beinhalten (i) 0,2 bis 5 Sekunden mittlere Verweilzeit, (ii) 15 bis 26 m/s Spitzengeschwindigkeit und (iii) 0,2 bis 3 kJ/kg Energiezustand, wobei Agglomerate gebildet werden; und
   (b) Granulieren der Agglomerate in einem Wirbelbett-Trockner und Wirbelbett-Kühler, wobei Bedingungen jeder der Fluidisierungsvorrichtungen 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 Fluidisierungsgeschwindigkeit und (vi) 12 bis 100 °C Betttemperatur, wobei die mittlere Verweilzeit in Schritt (b) insgesamt 2 bis 12 Minuten beträgt, und wobei eine Beschichtungsmittel, gewählt aus der Gruppe, bestehend aus Aluminosilicaten, Silicaten, Carbonaten und Mischungen hiervon zu Schritt (b)
gegeben wird, entweder direkt nach dem Wirbelbett-Kühler oder Wirbelbett-Trockner; und/oder zwischen dem Wirbelbett-Trockner und Wirbelbett-Kühler; und/oder direkt zu dem Wirbelbett-Trockner.

2. Verfahren nach Anspruch 1, wobei das Tensid aus der Gruppe gewählt wird, bestehend aus anionischem Tensid, nichtionischem Tensid, kationischem Tensid, zwittrionischem Tensid, ampholytischem Tensid und Mischungen hiervon.


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


7. Verfahren nach Anspruch 1, wobei ein interner Recyclatexstrom aus Pulver von der Fluidisierungsvorrichtung weiterhin zu Schritt (a) gegeben wird.

Revidierungen

1. Procédé sans tour de préparation d'une composition détergente granulaire ayant une masse volumique d'au moins 600 g/l, comprenant les étapes consistant à :
   (a) disperser un tensioactif, et revêtir le tensioactif d'une fine poudre ayant un diamètre de 0, 1 à 500 microns, en humidifiant le tensioactif revêtu de la fine poudre avec un liquide finement atomisé, dans un mélangeur dans lequel des conditions du mélangeur comprennent (i) 0,2 à 5 secondes de temps de séjour moyen, (ii) 15 à 26 m/s de vitesse périphérique, et (iii) 0,2 à 3 kJ/kg de condition d'énergie, dans lequel des agglomérats sont formés ; et
   (b) granuler les agglomérats dans un sécheur à lit fluidisé et un refroidisseur à lit fluidisé où les conditions de chacun des appareils de fluidisation comprennent (i) 1 à 10 minutes de temps de séjour moyen, (ii) 100 à 300 mm de profondeur de lit non fluidisé, (iii) pas plus de 50 microns de taille des gouttelettes de pulvérisation, (iv) 175 à 250 mm de hauteur de pulvérisation, (v) 0,2 à 1,4 m/s de vitesse de fluidisation et (vi) 12 à 100 °C de température de lit, où le temps de séjour moyen de l'étape (b) est au total 2 à 12 minutes et où un agent de refroidissement choisi dans le groupe constitué par des aluminosilicates, des silicates, des carbonates et leurs mélanges est ajouté à l'étape (b) directement après le refroidisseur à lit fluidisé ou le sécheur à lit fluidisé ; et/ou entre le sécheur à lit fluidisé et le refroidisseur à lit fluidisé ; et/ou directement au sécheur à lit fluidisé.

2. Procédé selon la revendication 1, dans lequel ledit tensioactif est choisi dans le groupe constitué par un tensioactif anionique, un tensioactif non ionique, un tensioactif cationique, un tensioactif zwittrionique, un tensioactif ampholyte et leurs mélanges.

3. Procédé selon la revendication 1, dans lequel ledit tensioactif est choisi dans le groupe constitué par les alkylbenzènesulfonates, les alkylsulfates alcoxyés, les éthoxylates d'alkyle, les alkylsulfates, les sulfates d'alcools gras de coprah et leurs mélanges.

4. Procédé selon la revendication 1, dans lequel on disperse une solution de polymère aqueuse ou non aqueuse avec ledit tensioactif dans l'étape (a).

5. Procédé selon la revendication 1, dans lequel la poudre fine est choisie dans le groupe constitué par le carbonate de soude, le tripolyposphat de sodium pulvérulent, le tripolyposphat hydraté, les sulfates sodiques, des alu-
minosilicates, des phyllosilicates cristallins, des phosphates, des silicates précipités, des polymères, des carbonates, des citrates, des nitrilotriacétates, des tensioactifs pulvérulents et leurs mélanges.

6. Procédé selon la revendication 1, dans lequel le liquide finement atomisé est choisi dans le groupe constitué par des silicates liquides, des tensioactifs anioniques, des tensioactifs cationiques, des solutions de polymères aqueuses, des solutions de polymères non aqueuses, l'eau et leurs mélanges.

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