



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) **EP 0 915 958 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention
of the grant of the patent:
04.06.2003 Bulletin 2003/23

(51) Int Cl.7: **C11D 11/00**, C11D 3/12,
C11D 17/06

(21) Application number: **97934907.3**

(86) International application number:
PCT/US97/11985

(22) Date of filing: **16.07.1997**

(87) International publication number:
WO 98/004669 (05.02.1998 Gazette 1998/05)

(54) **PREPARATION OF LOW IDENSITY DETERGENT AGGLOMERATES CONTAINING SILICA**

HERSTELLUNG VON KIESELSÄURE ENTHALTENDE WASCHMITTELAGGLOMERATE MIT
NIEDRIGER DICHTEN

PROCEDE DE FABRICATION D'AGGLOMERATS DETERGENTS DE FAIBLE DENSITE
CONTENANT DE LA SILICE

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

• **HUTCHINS, James, Peyton**
Cincinnati, OH 45224 (US)

(30) Priority: **26.07.1996 US 22816 P**

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

(43) Date of publication of application:
19.05.1999 Bulletin 1999/20

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

(56) References cited:
EP-A- 0 510 746 **WO-A-97/10321**
US-A- 5 576 285

(72) Inventors:
• **ROGERS, Steven, Barrett**
Cincinnati, OH 45247 (US)
• **FRANCE, Paul, Amaat**
West Chester, OH 45069 (US)

• **DATABASE WPI Week 9537 Derwent**
Publications Ltd., London, GB; AN 95-281206
XP002050465 & JP 07 179 898 A (LION CORP.) ,
18 July 1995

EP 0 915 958 B1

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).

DescriptionFIELD OF THE INVENTION

5 [0001] The present invention generally relates to a process for producing a low density detergent composition. More particularly, the invention is directed to a process during which low density detergent agglomerates are produced by feeding a surfactant paste and dry starting detergent material which includes a silica material into a high speed mixer followed by a drying apparatus. The process produces a free flowing, low density detergent composition which can be commercially sold as a conventional non-compact detergent composition or used as an admix in a low dosage, "compact" detergent product.

BACKGROUND OF THE INVENTION

15 [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. Consequently, there is a need in the art of producing modern detergent compositions for flexibility in the ultimate density of the final composition.

20 [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. 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. In both processes, the most important factors which govern the density of the resulting detergent granules are the density, shape, porosity and surface area of the various starting materials and their respective chemical composition. These parameters, however, can only be varied within a limited range. Thus, flexibility in the substantial bulk density can only be achieved by additional processing steps which lead to lower densities of the detergent granules.

25 [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 do not provide a process which has the flexibility of providing lower density granules.

30 [0005] 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.

35 [0006] 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 a starting detergent materials in the form of pastes, liquids and dry materials can be effectively agglomerated into crisp, free flowing detergent agglomerates having low densities.

40 [0007] Accordingly, there remains a need in the art to have a process for continuously producing a low density detergent composition directly from starting detergent ingredients. Also, there remains a need for such a process which is more efficient, flexible and economical to facilitate large-scale production of detergents of low as well as high dosage levels.

BACKGROUND ART

5 [0008] The following references are directed to densifying spray-dried granules: Appel et al, U.S. Patent No. 5,133,924 (Lever); Appel et al, U.S. Patent No. 5,164,108 (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. The following references are directed to producing detergents by agglomeration: 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); and Swatting et al, U.S. Patent No. 5,205,958. EP-A-0 510 746 relates to a process for producing condensed detergent particles.

SUMMARY OF THE INVENTION

15 [0009] The present invention meets the aforementioned needs in the art by providing a process which produces a low density (less than about 500 g/l) detergent composition directly from starting ingredients such as a surfactant paste and dry detergent ingredients. The dry ingredients include a silica material which ultimately is agglomerated such that it forms part of or is in the agglomerate particles themselves rather than "coated" on the outer surface of the agglomerates. Unexpectedly, this provides a low density agglomerate composition having improved physical properties. The process does not use the conventional spray drying towers currently used and is therefore 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.

20 [0010] As used herein, the term "agglomerates" refers to particles formed by agglomerating detergent granules or particles which typically have a smaller mean particle size than the formed agglomerates. All percentages used herein are expressed as "percent-by-weight" unless indicated otherwise. All viscosities described herein are measured at 70°C and at shear rates between about 10 to 50 sec⁻¹, preferably at 25 sec⁻¹.

25 [0011] In accordance with one aspect of the invention, a process for preparing low density detergent agglomerates having a density of below about 500 g/l is provided. The process comprises the steps of: (a) agglomerating a detergent surfactant paste and dry starting detergent material in a high speed mixer to obtain detergent agglomerates, wherein the dry starting detergent material includes a silica material; and (b) drying the detergent agglomerates so as to form the detergent composition having a density of less than 500g/l.

30 [0012] In accordance with another aspect of the invention, another process for preparing low density detergent agglomerates having a density of below about 500 g/l is provided. The process comprises the steps of: (a) agglomerating a detergent surfactant paste and dry starting detergent material in a high speed mixer to obtain detergent agglomerates, wherein the dry starting detergent material includes a fumed silica material; (b) mixing the detergent agglomerates in a moderate speed mixer to further agglomerate the detergent agglomerates; and (c) drying the detergent agglomerates so as to form the low density detergent composition having a density of less than about 500 g/l. The low density detergent composition made by any of the process embodiments described herein is also provided.

35 [0013] Accordingly, it is an object of the invention to provide a process for continuously producing a low density detergent composition directly from starting detergent ingredients. It is also an object of the invention to provide a process which is more efficient, flexible and economical to facilitate large-scale production of detergents of low as well as high dosage levels. 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

40 [0014] The present invention is directed to a process which produces free flowing, low density detergent agglomerates having a density of below about 500 g/l, preferably from about 350 g/l to about 500 g/l. The process produces low density detergent agglomerates from a highly viscous surfactant paste having a relatively high water content, typically at least about 10%. Generally speaking, the present process is used in the production of normal as opposed to low dosage detergents whereby the resulting detergent agglomerates can be used as a detergent or as a detergent additive. It should be understood that the process described herein can be continuous or batch depending upon the desired application.

Process

45 [0015] In the first step of the process, starting detergent materials are fed into a mixer for agglomeration. To achieve the desired density of below 500 g/l. the agglomeration step is carried forth initially in a high speed mixer after which

an optional moderate speed mixer may follow if further agglomeration is desired. The starting detergent materials preferably include a highly viscous surfactant paste and dry detergent material, the components of which are described more fully hereinafter. The starting detergent materials are agglomerated in the presence of a silica material as described more fully hereinafter to produce agglomerate particles having the desired low density. To that end, the instant process preferably entails mixing from about 1% to about 20%, more preferably from about 3% to about 10%, and most preferably from about 3% to about 5%, by weight of a silica material into the high speed mixer. The nature and composition of the entering or starting detergent materials and optional adjunct ingredients can vary as described in detail hereinafter.

[0016] It has unexpectedly been found that by having the silica in the agglomerate rather than as a coating agent, a low density agglomerate can be formed. This is contrary to expectations since using silica as a coating or dusting agent such that it is deposited on the outer surface of agglomerate particles will raise or increase the density. However, as demonstrated by the present process invention, silica inside the agglomerate particles lowers the density as is desired for producing a low density agglomerated composition. While not intending to be bound by theory, it is believed that the silica when subjected to agglomeration with a surfactant and subsequent drying step forms a hollow "finger-like" structure inside the individual agglomerate particles, thereby increasing the internal void space in each agglomerate particle. This, of course, results in more porous agglomerates having a relatively low density.

[0017] The other essential step in the process involves drying the agglomerates exiting the high speed mixer or moderate speed mixer if it is optionally used. This can be completed in a wide variety of apparatus including but not limited to fluid bed dryers. The drying step enhances the free flowability of the agglomerates and facilitates the "fluffy" or "puffy" physical characteristics of the resulting agglomerates. Accordingly, sufficient drying must occur in order to produce the desired low density agglomerates. In that regard, the drying temperature used in the whichever drying apparatus will preferably be from about 50 °C to about 300 °C, more preferably from about 80 °C to about 250 °C, and most preferably, from about 100°C to about 250°C.

[0018] Preferably, the mean residence time of the starting detergent materials in the high speed mixer (e.g. Lödige Recycler CB 30 or other similar equipment) is from about 2 to 45 seconds while the residence time in the optional low or moderate speed mixer (e.g. Lödige Recycler KM 300 "Ploughshare" or other similar equipment) is from about 0.5 to 15 minutes.

[0019] The detergent agglomerates produced by the process preferably have a surfactant level of from about 20% to about 55%, more preferably from about 35% to about 55% and, most preferably from about 45% to about 55%. The particle porosity of the resulting detergent agglomerates produced according to the process of the invention is preferably in a range from about 5% to about 50%, more preferably at about 25%. In addition, an attribute of dense or densified agglomerates is the relative particle size. The present process typically provides detergent agglomerates having a mean particle size of from about 250 microns to about 1000 microns, and more preferably from about 400 microns to about 600 microns. As used herein, the phrase "mean particle size" refers to individual agglomerates and not individual particles or detergent granules. The combination of the above-referenced porosity and particle size results in agglomerates having density values of below 500 g/l. Such a feature is especially useful in the production of laundry detergents having varying dosage levels as well as other granular compositions such as dishwashing compositions.

Optional Process Steps

[0020] In an optional step of the present process, the detergent agglomerates exiting the fluid bed dryer are further conditioned by cooling the agglomerates in a fluid bed cooler or similar apparatus as are well known in the art. Another optional process step involves adding a coating agent to improve flowability and/or minimize over agglomeration of the detergent composition 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 ; (2) the coating agent may be added between the fluid bed dryer and the fluid bed cooler, (3) the coating agent may be added between the fluid bed dryer and the moderate speed mixer, and/or (4) the coating agent may be added directly to the moderate speed mixer and 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 of detergent during use, but also serves to control agglomeration by preventing or minimizing over agglomeration, especially when added directly to the optional moderate speed mixer. 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.

[0021] Optionally, the process can comprise the step of spraying an additional binder in one or both of the mixer or fluid bed dryers. 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, 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.).

[0022] 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 in the one of the aforementioned drying apparatus.

[0023] Other optional steps in the present process involve recycling oversized and undersized agglomerates as described in Capeci et al, U.S. Patent Nos. 5,489,392 and 5,516,448 (Procter & Gamble). Also, the step of including an anhydrous material at selected points in the process can be incorporated as described by Capeci et al, U.S. Patent No. 5,366,652 and 5,486,303 (Procter & Gamble). Optionally, the agglomerates exiting the moderate speed mixer can be dried in a spray drying tower as described in Capeci et al, U.S. Patent 5,496,487 (Procter & Gamble).

[0024] 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.

Detergent Surfactant Paste

[0025] The detergent surfactant paste used in the process is preferably in the form of an aqueous viscous paste, although forms are also contemplated by the invention. This so-called viscous surfactant paste has a viscosity of from about 5,000 cps to about 100,000 cps, more preferably from about 10,000 cps to about 80,000 cps, and contains at least about 10% water, more preferably at least about 20% water. The viscosity is measured at 70°C and at shear rates of about 10 to 100 sec.⁻¹. Furthermore, the surfactant paste, if used, preferably comprises a deterative surfactant in the amounts specified previously and the balance water and other conventional detergent ingredients.

[0026] The surfactant itself, in the viscous surfactant paste, 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 3,919,678, 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.

[0027] Nonlimiting examples of the preferred anionic surfactants useful in the surfactant paste include the conventional C₁₁-C₁₈ alkyl benzene sulfonates ("LAS"), primary, branched-chain and random C₁₀-C₂₀ alkyl sulfates ("AS"), the C₁₀-C₁₈ secondary (2,3) alkyl sulfates of the formula CH₃(CH₂)_x(CHOSO₃⁻M⁺)CH₃ and CH₃(CH₂)_y(CHOSO₃⁻M⁺)CH₂CH₃ 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 C₁₀-C₁₈ alkyl alkoxy sulfates ("AE_xS"; especially EO 1-7 ethoxy sulfates).

[0028] Optionally, other exemplary surfactants useful in the paste of the invention include C₁₀-C₁₈ alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the C₁₀₋₁₈ glycerol ethers, the C₁₀-C₁₈ alkyl polyglycosides and their corresponding sulfated polyglycosides, and C₁₂-C₁₈ alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the C₁₂-C₁₈ alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C₆-C₁₂ alkyl phenol alkoxyates (especially ethoxylates and mixed ethoxy/propoxy), C₁₂-C₁₈ betaines and sulfobetaines ("sultaines"), C₁₀-C₁₈ amine oxides, and the like, can also be included in the overall compositions. The C₁₀-C₁₈ N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C₁₂-C₁₈ N-methylglucamides. See WO 9,206,154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C₁₀-C₁₈ N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C₁₂-C₁₈ glucamides can be used for low sudsing. C₁₀-C₂₀ conventional soaps may also be used. If high sudsing is desired, the branched-chain C₁₀-C₁₆ soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

Dry Detergent Material

[0029] The starting dry detergent material of the present process comprises a silica material. Silica is highly dispersed amorphous silicon dioxide that is commercially available in many forms. Most commonly, silica has a tapped density of from 50 g/l to 120g/l. The specific surface area of the particles ranges from 25 square meters per gram to 800 square meters per gram. The surface of the silica particles can be chemically modified to change their behavior with respect to water. For example, silica particles may be treated with organosilanes to make the particles predominantly hydrophobic. It has been found that silicas that are hydrophobic in nature work extremely well in the current process invention; however, hydrophilic silicas are useful in the process, as well. Additionally, diatomaceous earth materials may be used

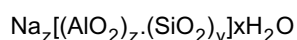
in conjunction with, or as a substitute for, the silicas mentioned herein.

[0030] Commercially, silicas are usually prepared by one of two techniques; either by precipitation (i.e. precipitated silicas) or by high temperature flame hydrolysis (i.e. fumed silicas). Both precipitated silicas and fumed silicas are useful in the current invention. Precipitated silicas generally have an agglomerate size of from 3 micrometers to 100 micrometers, whereas fumed silicas made by flame hydrolysis usually have particles which are substantially spherical and have an average particle diameter of from about 7 nm to about 40 nm. Fumed silicas having an average particle size of from about 7 to about 25 nm are preferred in the present invention. Exemplary commercially available silicas useful in the process invention include those supplied by Degussa AG (Germany) identified as Aerosil™, and particularly preferred is Aerosil™ R927. Silicas which are hydrophobic, fumed and have surface areas of at least about 110 square meters per gram and an average particle diameter size of 16 nm are most preferred.

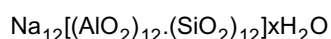
[0031] The dry detergent material also preferably includes a detergent aluminosilicate builder which are referenced as aluminosilicate ion exchange materials. The aluminosilicate ion exchange materials used herein as a detergent builder preferably have both a high calcium ion exchange capacity and a 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 inter-related 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).

[0032] Preferably, the aluminosilicate ion exchange material is in "sodium" form since the potassium and hydrogen forms of the instant aluminosilicate do not exhibit the 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.

[0033] Preferably, the aluminosilicate ion exchange material has the formula



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



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.

[0034] The aluminosilicates used herein are further characterized by their ion exchange capacity which is at least about 200 mg equivalent of CaCO₃ hardness/gram, calculated on an anhydrous basis, and which is preferably in a range from about 300 to 352 mg equivalent of CaCO₃ hardness/gram. Additionally, the instant aluminosilicate ion exchange materials are still further characterized by their calcium ion exchange rate which is at least 130mg Ca⁺⁺/litre/minute/gram/litre (2 grains Ca⁺⁺/gallon/minute/-gram/gallon), and more preferably in a range from 130mg Ca⁺⁺/litre/minute/gram/litre (2 grains Ca⁺⁺/gallon/minute/-gram/gallon) to 390mg Ca⁺⁺/litre/minute/gram/litre (6 grains Ca⁺⁺/gallon/minute/-gram/gallon).

[0035] An especially preferred set of dry detergent material is selected from the group consisting of aluminosilicates, crystalline layered silicates, sodium carbonate and mixtures thereof.

Adjunct Detergent Ingredients

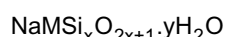
[0036] The starting dry detergent material in the present process can include additional detergent ingredients and/or, any number of additional ingredients can be incorporated in the detergent composition during subsequent steps of the present process. These adjunct ingredients include other detergency builders, bleaches, bleach activators, suds boosters or suds suppressers, anti-tarnish and anti-corrosion 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..

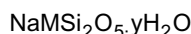
[0037] Other builders can be generally selected from the various water-soluble, alkali metal, ammonium or substituted ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, borates, polyhydroxy sulfonates, polyacetates, carboxylates, and polycarboxylates. Preferred are the alkali metal, especially sodium, salts of the above. Preferred for use herein are the phosphates, carbonates, C₁₀₋₁₈ fatty acids, polycarboxylates, and mixtures thereof. More preferred are sodium tripolyphosphate, tetrasodium pyrophosphate, citrate, tartrate mono- and di-succinates, and mixtures thereof (see below).

[0038] 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.

[0039] The crystalline layered sodium silicates suitable for use herein preferably have the formula



wherein M is sodium or hydrogen, x is from about 1.9 to about 4 and y is from about 0 to about 20. More preferably, the crystalline layered sodium silicate has the formula



wherein M is sodium or hydrogen, and y is from about 0 to about 20. These and other crystalline layered sodium silicates are discussed in Corkill et al, U.S. Patent No. 4,605,509.

[0040] 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.

[0041] Examples of nonphosphorus, inorganic builders are tetraborate decahydrate and silicates having a weight ratio of SiO₂ 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 acid.

[0042] 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, 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.

[0043] 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. 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.

[0044] 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..

[0045] 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.

[0046] 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 I-IV

[0047] These Examples illustrates a batch mode of the instant process. A low density agglomerated detergent composition is prepared using a lab tilt-a-pin (commercially available from Processall, Inc.) mixer. The mixer is first charged with a mixture of powders, namely sodium carbonate (mean particle size of 5-40 microns made via Air Classifier Mill), light density, granular sodium tripolyphosphate (commercially supplied by FMC Corp. and referenced herein as "STPP"), zeolite type A (commercially supplied by Ethyl Corp. and noted as below as "Zeolite A"), and fumed silica (commercially supplied by Cabot Corporation under the trade name M-5 Cab-o-sil™), except Example IV did not use this or any fumed silica material. A surfactant paste containing sodium linear alkylbenzene sulfonate ("LAS" as referenced herein) surfactant (75% by weight) and the balance water is then added on top of the powder mixture while the mixer is operated for about 15 seconds at 700 rpm until discrete granules are formed in the mixer. The composition of the agglomerates formed are set forth below in Table I.

TABLE I

| (% weight) | | | |
|-----------------------------|--------------|--------------|--------------|
| Component | I | II | III |
| LAS | 30.0 | 30.0 | 30.0 |
| Sodium carbonate (soda ash) | 36.0 | 36.0 | 34.0 |
| STPP | 29.0 | 29.0 | 29.0 |
| Zeolite A | 2.0 | 0.0 | 0.0 |
| Fumed silica | 3.0 | 5.0 | 7.0 |
| | <u>100.0</u> | <u>100.0</u> | <u>100.0</u> |
| Bulk Density (g/l) | 490 | 460 | 350 |

Unexpectedly, the resulting agglomerates of Examples I, II and II have a bulk density below 500 g/L, all of which are made by a process involving silica and within the scope of the process invention.

[0048] Having thus described the invention in detail, it will be clear 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 process for preparing a low density detergent composition character by the steps of:
 - (a) agglomerating a detergent surfactant paste and dry starting detergent material in a high speed mixer to obtain detergent agglomerates, wherein said dry starting detergent material includes a silica material; and
 - (b) drying said detergent agglomerates so as to form said detergent composition having a density of less than 500 g/l.
2. A process according to claim 1 wherein the density of said detergent composition is from 350 g/l to 500 g/l.
3. A process according to claims 1-2 wherein said dry starting material further comprises a builder selected from the group consisting of aluminosilicates, crystalline layered silicates, sodium carbonate and mixtures thereof.
4. A process according to claims 1-3 wherein said silica material is fumed silica.
5. A process according to claims 1-4 wherein said silica material is hydrophobic silica.
6. A process according to claims 1-5 wherein said silica material is hydrophilic silica.

7. A process according to claims 1-6 wherein said silica material is precipitated silica.
8. A process according to claims 1-7 further **characterized by** the step of mixing said detergent agglomerates in a moderate speed mixer after said agglomerating step and before said drying step to further agglomerate said detergent agglomerates.
9. A process according to claims 1-8 wherein said dry starting material further comprises diatomaceous earth materials.

10. A process for preparing a low density detergent composition **characterized by** the steps of:

(a) agglomerating a detergent surfactant paste and dry starting detergent material in a high speed mixer to obtain detergent agglomerates, wherein the dry starting detergent material includes a fumed silica material; mixing said detergent agglomerates in a moderate speed mixer to further agglomerate said detergent agglomerates; and drying said detergent agglomerates so as to form said low density detergent composition having a density of less than 500 g/l.

Patentansprüche

1. Verfahren zur Herstellung einer Reinigungsmittelzusammensetzung niedriger Dichte, **gekennzeichnet durch** die Schritte:

(a) Agglomerieren einer Reinigungsmittel-Tensidpaste und trockenes Ausgangsreinigungsmittelmateriale in einem Hochgeschwindigkeitsmischer, um Reinigungsmittelagglomerate zu erhalten, wobei das trockene Ausgangsreinigungsmittelmateriale ein Silicamateriale beinhaltet; und
(b) Trocknen der Reinigungsmittelagglomerate, um so die Reinigungsmittelzusammensetzung mit einer Dichte von weniger als 500 g/l zu bilden.

2. Verfahren nach Anspruch 1, wobei die Dichte der Reinigungsmittelzusammensetzung 350 g/l bis 500 g/l beträgt.

3. Verfahren nach den Ansprüchen 1-2, wobei das trockene Ausgangsmateriale weiterhin einen Builder umfaßt, gewählt aus der Gruppe, bestehend aus Aluminosilicaten, kristallinen Schichtsilicaten, Natriumcarbonat und Mischungen hiervon.

4. Verfahren nach den Ansprüchen 1-3, wobei das Silicamateriale Quarzstaub ist.

5. Verfahren nach den Ansprüchen 1-4, wobei das Silicamateriale hydrophobes Silica ist.

6. Verfahren nach den Ansprüchen 1-5, wobei das Silicamateriale hydrophiles Silica ist.

7. Verfahren nach den Ansprüchen 1-6, wobei das Silicamateriale ausgefälltes Silica ist.

8. Verfahren nach den Ansprüchen 1-7, weiterhin **gekennzeichnet durch** den Schritt des Mischens der Reinigungsmittelagglomerate in einem Mäßiggeschwindigkeitsmischer nach dem Agglomerierungsschritt und vor dem Trocknungsschritt, um die Reinigungsmittelagglomerate weiter zu agglomerieren.

9. Verfahren nach den Ansprüchen 1-8, wobei das trockene Ausgangsmateriale weiterhin Diatomeenerde-Materiale umfaßt.

10. Verfahren zur Herstellung einer Reinigungsmittelzusammensetzung niedriger Dichte, **gekennzeichnet durch** die Schritte:

(a) Agglomerieren einer Reinigungsmittel-Tensidpaste und trockenes Ausgangsreinigungsmittelmateriale in einem Hochgeschwindigkeitsmischer, um Reinigungsmittelagglomerate zu erhalten, wobei das trockene Ausgangsreinigungsmittelmateriale ein Quarzstaubmateriale umfaßt;
(b) Mischen der Reinigungsmittelagglomerate in einem Mäßiggeschwindigkeitsmischer, um die Reinigungs-

mittelagglomerate weiter zu agglomerieren; und

(c) Trocknen der Reinigungsmittelagglomerate, um so die Reinigungsmittelzusammensetzung niedriger Dichte mit einer Dichte von weniger als 500 g/l zu bilden.

5

Revendications

1. Procédé de préparation d'une composition détergente basse densité, **caractérisé par** les étapes consistant à :

- 10 (a) agglomérer une pâte tensioactive détergente et une matière détergente de départ sèche dans un mélangeur à vitesse élevée pour obtenir des agglomérats détergents, où ladite matière détergente de départ sèche comprend une matière siliceuse ; et
(b) sécher lesdits agglomérats détergents de manière à former ladite composition détergente ayant une densité inférieure à 500 g/l.

15 2. Procédé selon la revendication 1, dans lequel la densité de ladite composition détergente est de 350 g/l à 500 g/l.

20 3. Procédé selon les revendications 1 - 2, dans lequel ladite matière de départ sèche comprend en outre un adjuvant choisi dans le groupe constitué par des aluminosilicates, des silicates lamellaires cristallins, du carbonate de sodium et leurs mélanges.

4. Procédé selon les revendications 1 - 3, dans lequel ladite matière siliceuse est la silice filmée.

25 5. Procédé selon les revendications 1 - 4, dans lequel ladite matière siliceuse est la silice hydrophile.

6. Procédé selon les revendications 1 - 5, dans lequel ladite matière siliceuse est la silice hydrophile.

7. Procédé selon les revendications 1 - 6, dans lequel ladite matière siliceuse est la silice précipitée.

30 8. Procédé selon les revendications 1 - 7, **caractérisé en outre par** l'étape consistant à mélanger lesdits agglomérats détergents dans un mélangeur à vitesse modérée après ladite étape d'agglomération et avant ladite étape de séchage pour agglomérer davantage lesdits agglomérats détergents.

35 9. Procédé selon les revendications 1 - 8, dans lequel ladite matière de départ sèche comprend en outre des matières de terre diatomée.

10. Procédé de préparation d'une composition détergente basse densité, **caractérisé par** les étapes consistant à :

- 40 (a) agglomérer une pâte tensioactive détergente et une matière détergente de départ sèche dans un mélangeur à vitesse élevée pour obtenir des agglomérats détergents, où ladite matière détergente de départ sèche comprend une matière de silice fumée ;
(b) mélanger lesdits agglomérats détergents dans un mélangeur à vitesse modérée pour agglomérer davantage lesdits agglomérats détergents ; et
(c) sécher lesdits agglomérats détergents de manière à former ladite composition détergente basse densité
45 ayant une densité inférieure à 500 g/l.

50

55