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(54) Laundry detergent composition and process

(57) Detergent powder manufacturing process in which solid alkali particulate material is blended and reacted with liquid anionic acid surfactant precursor in a mixer to form anionic surfactant and the solid particulate alkali is also blended with nonionic surfactant added in a liquid form to the same mixer, characterised in that the nonionic surfactant is added in admixture with anionic surfactant which has been neutralised using a liquid alkaline solution. In addition, the powder produced according to the process and tablets made by compressing the powder, optionally in admixture with other detergent ingredients. In addition, use of the tablets so formed in the laundering of fabrics.

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Description

Field of the Invention

[0001] This invention relates to a mixing process to manufacture detergent powder that contains anionic surfactant, to detergent compositions containing the powder and to tablets made by compressing the powder compositions and their use in fabric washing.

Background to the Invention

[0002] Laundry Detergent compositions in tablet form are available. Often these tablets are manufactured from powder formulations.

[0003] High bulk density detergent powders produced via mechanical granulation routes, so called 'non-tower granulation routes', are well known. These concentrated base powders can be used in powder formulations, which are then converted into tablets, or parts thereof, by any suitable process, for example by compression.

[0004] A non-tower granulation route is typically not designed to produce base powders suitable for manufacture of tablets. Consequently, problems have been encountered when acid forms of anionic surfactant are neutralised to make base powders comprising surfactant granules. If the neutralisation is not fully complete at the time the granules are further processed into tablets the quality and properties of the tablets can be adversely affected.

[0005] Known non-tower granulation routes typically consist of at least one mechanical mixer. For example, the non-tower route described in EP-A-367339 (Unilever) consists of a high shear mechanical mixer for agglomerating fine particulate solids with a binder to form a granular powder, followed by a medium shear mechanical mixer for densification of the granules in the powder and a fluidised bed for drying and/or cooling the product. Other non-tower routes can, for example, comprise only a single batch medium-shear mixer, for example a ploughshare mixer. Granular concentrated base powders produced via non-tower routes usually have a bulk density of at least 650 grams per litre.

[0006] Non-tower granulation routes are known to be suitable for producing concentrated base powders via dry neutralisation of acid precursors of anionic surfactants, such as alkylbenzene sulphonic acids, by particulate solid materials comprising an alkaline source, such as sodium carbonate (JP application 60 072 999, Kao and EP-A-420317, Unilever). The acid precursor of the anionic surfactant and its neutralised reaction product, the surfactant itself, act as binders in the granules made by the process. Alternatively, nonreactive binders can be used such as mixtures of neutralised anionic and nonionic surfactants, either in liquid form (Colgate's EP-A-345090, Unilever's EP-A-265203 and EP-A-925354) or paste form (EP-A-656825 and EP-A-858500, Procter & Gamble). These binders can be either sprayed or pumped into a mechanical mixer.

[0007] Non-tower routes have been modified in a number of ways. For example: layering has been applied in the densification step of a continuous non-tower route in order to control the particle size of the granules (Unilever's EP-A-390251). In addition, shaft vibration was reduced to enable non-tower granulation with highly viscous binder pastes (EP-A-918843, P&G) .

[0008] A problem with the prior art non tower granulation processes is that if concentrated base powders are produced that are optimised for properties so that they can be included in formulations in powder form then they are not optimised for the properties of tablet formulations in which they may be included; especially where those tablets are obtained via processes comprising compression. It is therefore an object of the invention to provide a process for producing concentrated powders, which are especially optimised for use in formulations in tablet form, while retaining the necessary properties for their use in fully formulated powders.

Summary of the Invention

[0009] According to a first aspect of the present invention there is provided a detergent powder manufacturing process in which solid alkali particulate material is blended and reacted with liquid anionic acid surfactant precursor in a mixer to form anionic surfactant and the solid particulate alkali is also blended with nonionic surfactant added in a liquid form to the same mixer, characterised in that the nonionic surfactant is added in admixture with anionic surfactant which has been neutralised using a liquid alkaline solution.

[0010] Advantageously, the solid alkali is blended with the nonionic surfactant liquid before it is blended with the liquid anionic surfactant acid precursor. The solid alkaline material is preferably sodium carbonate.

[0011] Desirably, the anionic surfactant made by reaction with the solid alkali is the same as that made by reaction with the liquid alkaline solution. Preferably, the anionic surfactant is LAS.

[0012] According to a second aspect of the invention there is provided a powder detergent formulation including at least 20% of the powder formed according to the inventive process in admixture with up to 80% of granules selected from the list comprising, bleach, bleach activator, builder, soil release agents, coloured speckles, buffer, and other detergent formulation ingredients as defined herein.

[0013] According to a third aspect of the invention there is provided a tablet or discrete region thereof formed by compression of the powder according to the invention, as produced by the process of the invention. The tablets are used in laundering of clothes.

[0014] A particularly useful powder composition according to the invention is a granular detergent powder with a bulk density of more than 650 g/l obtained by the continuous process of:

a. Forming agglomerates from a finely dispersed powder comprising an alkali source in a high speed mechanical mixer by the addition of two separate liquid binders, in which:

- one liquid binder is a neutralised mixture of anionic and nonionic surfactants and 0.1-10% water, and
- the other liquid binder is an acid precursor of an anionic surfactant

b. Densification of said agglomerates in a medium speed mechanical mixer while adding 0.1-10% of a finely dispersed powder, preferably sodium aluminiumsilicate, and

c. Drying and/or cooling said agglomerates in a fluidised bed mixer.

[0015] The binder comprising the neutralised mixture of anionic and nonionic surfactants and 0.1-10% water may comprise up to 10 wt% soap.

[0016] We have found that in order to provide optimum properties for use in products produced by processes comprising a compressing step, such as formulations in tablet form, the individual components within the granules of the concentrated powder produced by a non-tower mechanical granulation process should be homogeneously distributed throughout these granules.

[0017] Without wishing to be bound by theory it is believed that this ensures that the granules provide the same binding force in any compressing step irrespective of their position relative to each other and to any other particulate components that are present in the formulation.

[0018] We have determined that granules produced by prior art non-tower granulation processes are not fully homogeneous, especially when one or more pastes are used as the binder(s), or when a liquid binder is converted to a soft solid, often sticky, or a viscous paste during one of the granulation steps. When a concentrated powder is to be used in compressed formulations, a binder that is a paste or a binder converted to a soft solid or viscous paste during granulation is preferred over a low-viscosity binder. This is because it provides more strength to the granules than a low-viscosity binder, which is probably due to the low viscosity binder being too easily squeezed out of the granules during their compression. However, low viscosity binders are known to enable easy mixing of the components in a non-tower granulation process. The present invention provides the advantages of both low and high viscosity binders by a novel combination of these binder types.

[0019] As stated above, we have found that the homogeneity of particles in concentrated base powders produced by known processes is not ideal. It is known from EP-466484 and EP-A-522766, that additional process steps are needed for concentrated granulated base powders produced via the prior art processes, when such powders are incorporated in compressed tablets. Examples of such additional process steps are: the removal of fines (<200 micron) by sieving, and/or coating the base powder or the other materials in tablets containing it with binder and/or disintegrant.

[0020] It is an object of the present invention to provide a process using non-tower mechanical granulation to produce concentrated detergent powders that are more homogeneous than those concentrated powders produced by prior methods and as a result give rise to superior properties of compressed products in which they may be incorporated. The concentrated powders of the invention are especially suitable for use in fabric washing formats that are produced via methods comprising a compression step, such as the tablet format, but can also be included in fabric washing products in powder form.

[0021] A fabric washing formulation comprising a concentrated granulated powder produced using the inventive process, not only provides a low variation in any subsequent process comprising compression, due to the individual granules having similar mechanical properties and surface compositions, but it also enables the use of lower compression forces. The higher level of homogeneity of the individual granules produced according to the invention enables optimisation of the composition of the concentrated powder in order to provide high binding strengths at relatively low compression forces. Because of this, residues obtained from compressed products, especially tablets, comprising the concentrated powder of the invention, are significantly reduced compared to the same compressed products comprising concentrated powders produced via the prior art processes.

[0022] Although particularly suitable for use in tablets, the concentrated powders of the invention can also be included in fabric washing formulations in powder form and in all unit dose formats comprising uncompressed or compressed powder, such as sachets or capsules.

[0023] Surprisingly, the concentrated base powders produced according to the process of the invention also exhibited improved dissolution compared with powders produced according to prior art processes. Without wishing to be bound by theory, it is believed that the improved degree of mixing of the solids and the surfactants in the process results in this unexpected advantage.

Detailed Description of the Invention

[0024] The process of the invention comprises mechanical granulation in one or more mechanical mixers of one or more fine solid particulate components comprising at least one alkali source with two liquid binders. The first liquid binder is a neutralised mixture of surfactants comprising at least one anionic surfactant. The second liquid binder is an acid precursor of an anionic surfactant, which reacts with the solid alkaline particulate component or components. The binders can be added simultaneously or sequentially into the mixer. One or both binders can be added at the same time as the solid particulate components, however it is preferred to add them after the solids have been added. The granulation process itself can be either a batch process or a continuous process.

Examples of mechanical mixers that can be used are:

[0025] As high-speed mixer/densifier may be used a Lödige (trademark) CB 30 Recycler. This apparatus essentially consists of a large, static hollow cylinder having a diameter of about 30 cm, which is horizontally placed. In the middle, it has a rotating shaft with several different types of blades mounted thereon. It can be rotated at speeds between 100 and 2500 rpm, dependent on the bulk density and the particle size desired. The blades on the shaft provide a thorough mixing action of the solids and the liquids, which may be admixed at this stage. The mean residence time is somewhat dependent on the rotational speed of the shaft, the position of the blades and the weir at the exit opening.

[0026] Other types of high-speed mixers/densifiers having a comparable effect on detergent powders can also be contemplated. For instance, a Shugi (trademark) Granulator or a Drais (trademark) K-TTP 80 may be used.

[0027] A second processing step can be successfully carried out in a medium shear mixer such as a Lödige (trademark) Ploughshare mixer. This apparatus essentially consists of a hollow static cylinder having a rotating shaft in the middle. On this shaft, various plough-shaped blades are mounted. It can be rotated at a speed of 40-160 rpm. Optionally, one or more high-speed cutters can be used to prevent excessive agglomeration. Another suitable machine for this step is, for example, the Drais (trademark) K-T 160.

Solid Components

[0028] Solid components that can be used in non-tower processes are well known. These solid components are often particulate solids including zeolites, salts such as sodium tripolyphosphate (STP), sodium carbonate, sodium sulphate and many other components which are usually dosed at a lower level than the aforementioned solid components, such as for example sodium acetate, trisodium citrate, alkali metal silicates and SCMC. As the current invention involves dry neutralisation of an acid precursor of an anionic surfactant in a non-tower process, it is essential that at least one of the solid components comprises an alkali source, for example fine sodium carbonate (soda ash).

[0029] In a preferred embodiment of the invention, the solid components comprise 20-90 wt%, preferably 40-80 wt%, more preferably 50-75 wt% of a zeolite, preferably zeolite MAP (these percentages include any water present within the zeolites), 10-30 wt% alkali carbonate, preferably sodium carbonate and 0-20 wt% of other solid components.

First Liquid Binder

[0030] The use of neutralised liquid surfactant mixtures comprising at least one anionic surfactant as binders in non-tower processes is well known. Examples of these mixtures can be found in EP-A-345090 (Colgate), EP-A-265203, EP-A-544365 and EP-A-925354 (Unilever).

[0031] In order to enable their use in the process of the current invention, it is a requirement for the aforementioned surfactant mixtures to have a low viscosity at the temperature which they are added to the mixer and a much higher viscosity when they are cooled to room temperature (25°C) .

[0032] In order to obtain homogeneous granules, neutralised surfactant mixtures can be used which have a viscosity of less than 2 Pa.s (measured at a shear rate of $50s^{-1}$ at temperatures of 40-100°C) when they enter the mixer or mixers and a viscosity of more than 5 Pa.s (measured at a shear rate of $50s^{-1}$) at room temperature (25°C).

Second Liquid Binder

[0033] The second liquid binder in the process of the invention is an acid precursor of an anionic surfactant. Well-known precursors in the field are alkylbenzene sulphonic acids, especially linear alkylbenzene sulphonic acid (LAS acids), and the acid forms of alkyl sulphates, especially acids of primary alkyl sulphates (PAS acids).

[0034] In order to obtain good dispersion of the first and second liquid binders and the reaction product of the acid precursor of an anionic surfactant and the solid alkali source throughout the granules, both binders must retain a sufficiently low viscosity during the granulation.

[0035] The temperature of the neutralised mixture of surfactants should remain high for as long as possible during the granulation stage in order to retain its low viscosity for ease of mixing. For that purpose, the temperature of the second liquid binder, the acid surfactant precursor, should be high enough to prevent it from increasing the viscosity of the neutralised mixture of surfactants too early in the granulation stage. The heat which is generated by the exothermic reaction of the second liquid binder with the alkaline solid materials enhances the mixing action by delaying the cooling and thus delaying the solidification or viscosity increase of the first liquid binder during the mixing stage. On the other hand, the temperature of the reactive second liquid binder should also be low enough to prevent excessive heat formation in the process during its neutralisation with the alkaline solid components.

Excessive temperature results in sticky powder.

[0036] Furthermore, when a sequential dosing of the two liquid binders is used, both the dosing rates of the binders and the time between the addition of the binders influence the binder viscosity during the granulation stage and therefore the level of homogeneity of the concentrated powder that is produced. As a result, for each given non-tower process, composition and mode of liquid binder addition the temperatures and dosing rates of the two binders used in the process may be balanced in such a way that optimum mixing is obtained. It has been found that the energy input of the mechanical mixer is a good indicator for monitoring how well the components are being mixed. The energy input decreases when the binders in the process become less viscous. As a consequence, a high level of homogeneity is obtained in the granules of the concentrated powder when the powder is produced at the lowest possible energy input for the non-tower process used - and when the powder at the same time contains low, for instance less than 5 wt%, levels of fine particles (smaller than 180 microns) and oversize material (larger than 2 mm).

[0037] In an especially preferred embodiment of the invention, the concentrated detergent powder is produced via a continuous granulation process comprising a high-speed mechanical mixer to agglomerate the fine particulate solid materials with both binders and a medium-speed mechanical mixer to densify, and optionally to layer, the granules. The final step in this process is cooling and/or drying of the resulting granular powder to ambient temperature in a fluidised bed mixer/dryer.

[0038] In this embodiment, the two liquid binders are dosed simultaneously into the continuous high-speed mechanical mixer. The temperature at which the first liquid binder is pumped into this mixer is usually not higher than 100°C, preferably not higher than 95°C, more preferably not higher than 90°C and not lower than 40°C, preferably not lower than 60°C, more preferably not lower than 80°C. The temperature of the second liquid binder is usually not higher than 90°C, preferably not higher than 80°C, more preferably not higher than 70°C and not lower than 30°C, preferably not lower than 40°C, more preferably not lower than 55°C. However, the optimal temperatures for both binders are highly dependent on their composition. For example, it is preferred that the temperature of primary alkyl sulphates does not exceed 60°C, in order to prevent their decomposition.

[0039] The concentrated powders produced via the process of the invention have a bulk density of more than 500 g/l, preferably more than 650 g/l and more preferably more than 750 g/l. They are flee-flowing and have a mean particle size of between 300 and 1200 μm, preferably between 400 and 1000 μm, more preferably between 450 and 750 μm. With the preferred process of the invention, bulk densities between 650 and 1200 g/l, preferably between 700 and 1000 g/l, more preferably between 750 and 900 g/l are obtained.

[0040] The concentrated powder produced by the process according to the invention may be used as a base for a powder formulation or it may be incorporated into a tablet via a compression process. In both instances, it is preferable that other materials are admixed with the powder during further processing steps. Suitable materials, which may be used in powders and tablets comprising the concentrated powder produced by the process of this invention, will now be discussed in more detail.

[0041] Weight percentages herein are calculated based upon total weight of the formulated product (powder, tablet, or other format), unless otherwise indicated.

Surfactant Compounds

[0042] In addition to surfactants already present in the concentrated powder produced according to the invention further surfactants selected from the group comprising anionic (soap or non-soap), cationic, zwitterionic, amphoteric, nonionic or a combination of these may be used.

[0043] For a tablet, these preferably provide from 5 to 50wt% of the overall tablet composition, more preferably from 8 or 9wt% of the overall composition up to 40% or 50wt%.

[0044] Anionic surfactant may be present in an amount from 0.5 to 50wt%, preferably from 2wt% or 4wt% up to 30wt%

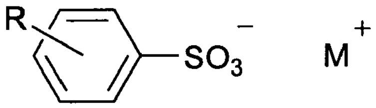
or 40wt% of a tablet composition.

[0045] Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

[0046] Primary alkyl sulphate having the formula

$$R\text{-}OSO_3^- M^+$$

in which R is an alkyl or alkenyl chain of 8 to 18 carbon atoms especially 10 to 14 carbon atoms and M^+ is a solubilising cation, is commercially significant as an anionic surfactant. Linear alkyl benzene sulphonate of the formula



where R is linear alkyl of 8 to 15 carbon atoms and M^+ is a solubilising cation, especially sodium, is also a preferred anionic surfactant.

[0047] Frequently, such linear alkyl benzene sulphonate or primary alkyl sulphate of the formula above, or a mixture thereof will be the desired anionic surfactant and may provide 75 to 100 wt% of any anionic soap surfactant in the composition.

[0048] The amount of non-soap anionic surfactant preferably lies in the range from 5 to 20 wt% of the total powder or tablet composition.

[0049] Soaps for use in accordance to the invention are preferably alkali metal or alkaline earth metal salts of naturally occurring fatty acids, preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil. Especially preferably, soaps are selected from C_{10} to C_{20} soaps for example from C_{16} to C_{18} or C_{12} soaps.

[0050] Suitable nonionic surfactant compounds which may be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide.

[0051] Specific nonionic surfactant compounds are alkyl (C_{8-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-diamine.

[0052] Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{9-11} and C_{12-15} primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

[0053] In preferred fabric washing tablets and powders comprising the powder of this invention, the amount of nonionic surfactant lies in a range from 4 to 40 wt%, more preferably 4 or 5 to 30 wt% of the total composition.

[0054] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition, optionally in admixture with other liquid components of the composition.

Detergency Builder

[0055] Powders and tablets comprising the concentrated powder of the invention will usually contain from 5 to 80 wt%, more usually 15 to 60 wt% of detergency builder. This may be provided wholly by water-soluble materials, or may be provided in large part or even entirely by water-insoluble material with water-softening properties. Water-insoluble detergency builder may be present as 5 to 80 wt%, better 5 to 60 wt% of the composition.

[0056] Alkali metal aluminosilicates are strongly favoured as environmentally acceptable water-insoluble builders for fabric washing. Alkali metal (preferably sodium) aluminosilicates may be either crystalline or amorphous or mixtures thereof, having the general formula:

$$0.8 - 1.5\ Na_2O.Al_2O_3.\ 0.8 - 6\ SiO_2.\ xH_2O$$

[0057] These materials contain some bound water (indicated as "xH_2O") and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as described in the literature.

[0058] Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in

GB 1429143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well known zeolites A and X; zeolite P described in EP 384070 (Unilever) and mixtures thereof.

[0059] A suitable water-insoluble detergency builder may be provided by a layered sodium silicate as described in US 4664839. NaSKS-6 is the trademark for a crystalline layered silicate marketed by Clariant (commonly abbreviated as "SKS-6"). NaSKS-6 has the delta-Na_2SiO_5 morphology form of layered silicate. It can be prepared by methods such as described in DE-A-3,417,649 and DE-A-3,742,043. Other such layered silicates, such as those having the general formula $NaMSi_xO_{2x+1}.yH_2O$ wherein M is sodium or hydrogen, x is a number from 1.9 to 4, preferably 2, and y is a number from 0 to 20, preferably 0 can be used.

[0060] Suitable water-soluble phosphorous-containing inorganic detergency builders, include the alkali-metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates. Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, orthophosphates and hexametaphosphates.

[0061] Non-phosphorous water-soluble builders used may be organic or inorganic. Inorganic builders that may be present include alkali metal (generally sodium) carbonate; while organic builders include polycarboxylate polymers, such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphonates, monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono- di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates and hydroxyethyliminodiacetates.

[0062] Fabric washing powders and tablets comprising the concentrated powder produced according to the inventive process also preferably include polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers which can function as builders and also inhibit unwanted deposition onto fabric from the wash liquor.

Bleach System

[0063] Tablets and powder formulations comprising the concentrated powder according to the invention may contain a bleach system preferably comprising one or more peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with activators to improve bleaching action at low wash temperatures. If any peroxygen compound is present, the amount is likely to lie in a range from 10 to 25 wt% of the composition.

[0064] Preferred inorganic persalts are sodium perborate monohydrate, tetrahydrate, and sodium percarbonate, advantageously employed together with an activator. Bleach activators, also referred to as bleach precursors, have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example, tetraacetylethylene diamine (TAED), now in widespread commercial use in conjunction with sodium perborate; and perbenzoic acid precursors. The quaternary ammonium and phosphonium bleach activators disclosed in US 4751015 and US 4818426 (Lever Brothers Company) are also of interest. Another type of bleach activator which may be used, but which is not a bleach precursor, is a transition metal catalyst as disclosed in EP-A-458397, EP-A-458398 and EP-A-549272. A bleach system may also include a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

[0065] As indicated above, if bleach is present as a water-soluble inorganic peroxygen bleach, the amount may be from 10 wt% to 25 wt% of the composition.

Other Detergent Ingredients

[0066] The detergent powders and tablets comprising the concentrated powder of the invention may also contain one or more of the detergency enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains. Suitable enzymes include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark. Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in amount of from about 0.1% to about 3.0% by weight of the composition; and these granules or marumes present no problems with respect to compression to form a tablet.

[0067] The detergent powders and tablets comprising the concentrated powder of the invention may also contain a fluorescer (optical brightener), for example, Tinopal (trademark) DMS or Tinopal CBS available from Ciba-Geigy AG, Basel, Switzerland. Tinopal DMS is disodium 4,4'bis-(2-morpholino-4-anilino-s-triazin-6-ylamino) stilbene disulphonate; and Tinopal CBS is disodium 2,2'-bis-(phenylstyryl) disulphonate.

[0068] An antifoam material is advantageously included; especially if a detergent powder or tablet is primarily intended for use in front-loading drum-type automatic washing machines. Suitable antifoam materials are usually in granular form, such as those described in EP 266863A (Unilever). Such antifoam granules typically comprise a mixture of silicone oil, petroleum jelly, hydrophobic silica and alkyl phosphate as antifoam active material, absorbed onto a porous absorbed water-soluble carbonate-based inorganic carrier material. Antifoam granules may be present in an amount of up to 5 wt% of the composition.

[0069] It may also be desirable that a detergent powder or tablet comprising the concentrated powder of the invention includes an amount of an alkali metal silicate, particularly sodium ortho-, meta- or di-silicate. The presence of such alkali metal silicates at levels, for example, of 0.1 to 10 wt%, may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some measure of building and giving processing benefits in manufacture of tablets.

[0070] A powder or tablet for fabric washing will generally not contain more than 15 wt% silicate.

[0071] Further ingredients which can optionally be in powders and tablets comprising the concentrated powder include antiredeposition agents such as sodium carboxymethylcellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose, fabric-softening agents; heavy metal sequestrants such as EDTA; perfumes; and colorants or coloured speckles.

[0072] Preferably tablets will comprise a disintegrant, more preferably a swellable disintegrant. A disintegrant is known in the art to aid disintegration and quick dissolution of the tablet in water. Suitable examples of swellable disintegrants are Acusol 771 (TM of Rohm & Haas), Disintex 75 (TM of ISP) or Arbocel (TM of Rettenmaier und Sohne). Disintegrant is suitably incorporated into a tablet at a level of more than 0.5 wt%.

[0073] Further ingredients, which can optionally be used in tablets incorporating the concentrated powder of the invention, are dispersing aids. Examples of suitable dispersing aids are water-swellable polymers (e.g. SCMC) highly soluble materials (e.g. sodium citrate, potassium carbonate or sodium acetate) or sodium tripolyphosphate with preferably at least 40% of the anhydrous phase I form.

[0074] Tabletting machinery able to carry out the manufacture of tablets includes suitable tablet presses available from Fette and from Korsch.

[0075] Tabletting may be carried out at ambient temperature or at a temperature above ambient which may allow adequate strength to be achieved with less applied pressure during compression. In order to carry out the tabletting at a temperature that is above ambient, the particulate composition is preferably supplied to the tabletting machinery at an elevated temperature. This will of course supply heat to the tabletting machinery, but the machinery may be heated in some other way also.

[0076] The size of a tablet will suitably range from 10 to 160 grams, preferably from 15 to 60 g, depending on the conditions of intended use, and whether it represents a dose for an average load in a fabric washing or dishwashing machine or a fractional part of such a dose. The tablets may be of any shape. However, for ease of packaging they are preferably blocks of substantially uniform cross-section, such as cylinders or cuboids. The overall density of a tablet preferably lies in a range from 1040 or 1050g/litre up to 1600g/litre.

[0077] The present invention will now be further described by way of the following non-limiting examples.

Methods

[0078] The dissolution time of a powder was determined by conductivity: A 600 ml glass beaker with a diameter of 85 mm was filled with 500 ml of demineralised water at 20°C. The water was stirred with a magnetic stirrer bar of 63 mm length while maintaining a vortex of 25 mm. A conductivity probe connected to an electronic datalogger was placed in the beaker near to the wall of the beaker. Then, 1.25 g of the powder was poured into the water. The dissolution time of the powder was recorded as the time after powder addition at which the conductivity had reached 90% of its maximum value.

[0079] The particle size of the powders was determined with a Retsch Camsizer.

[0080] Tablet strength was tested with a Synergy 100 materials testing machine manufactured by MTS Systems, France. The strength of a tablet may be expressed as F_{max}, which is measured by compressing the tablet radially, between the platens of a materials testing machine, until fracture of the tablet occurs. F_{max} in Newton is the force applied at fracture. The diametral fracture stress (DFS) of the tablet is then calculated using the equation:

$$\mathrm{DFS} = 2F_{max}/\pi Dt$$

where DFS is the diametral fracture stress in Pascal, D is the tablet diameter in metres and t is the tablet thickness in metres.

Examples

Materials used in the Examples

[0081] Zeolite MAP - Doucil A24(TM), supplied by Ineos Silicas. SCMC - Sodium carboxymethylcellulose, supplied

by AKZO. Sodium carbonate - Light soda ash, supplied by Brunner-Mond. Sodium acetate - Sodium acetate trihydrate supplied by Verdugt, to which 1% by weight zeolite MAP was added to improve its handling characteristics.
LAS acid - Linear alkylbenzene sulphonic acid, 97.1% pure, produced and supplied by the applicants.
Nonionic 7EO - Neodol(TM) 25-7E, supplied by Shell Chemicals.
Fatty acid - Pristerene(TM) 4916, supplied by Uniqema. 50% NaOH - 50% by weight aqueous solution of sodium hydroxide supplied by Chemproha.

Example 1

[0082] Solid materials (A) detailed in Table 1 were dosed continuously in a Lödige CB100 Recycler mixer operating at 1500 rpm. A liquid neutralised surfactant mixture (B) was produced separately in an in-line mixing system and pumped into the CB100 mixer at 90°C.

[0083] Simultaneously with mixture (B), LAS acid (C) was pumped into the CB100 mixer at 60°C. The product exiting the CB100 mixer was continuously fed into a Lödige KM10000 ploughshare mixer operating at 450rpm. Zeolite MAP was also fed to the ploughshare mixer for layering the product (D).

[0084] The resulting concentrated powder was then transported to a fluidised bed in which it was cooled to ambient temperature (the temperature of which varied during the process, but was always between 25 and 37°C) using dehumidified air at 10°C. Finally, coarse material larger than 2000 microns was removed from the product by sieving. The resulting product was a free-flowing concentrated base powder (E) with a bulk density of between 775 and 800 g/l and a mean particle size of between 450 and 600 microns. The total throughput of the process was 18000 kilograms per hour. Table 1 lists the weight percentages of the materials that were dosed into the process.

Table 1: Raw material flows in the process

Dosed in CB100 mixer		Wt%
Solid components (A)	Zeolite MAP	48.26
	SCMC	0.92
	Sodium carbonate	8.77
	Sodium acetate	5.83
Liquid neutralised surfactant mixture (B)	LAS acid	10.65
	Nonionic 7EO	9.47
	Fatty acid	1.46
	50% NaOH	3.00
Acid precursor of anionic surfactant (C)	LAS acid	9.53
Dosed in KM 10000 mixer		
layering (D)	Zeolite MAP	2.12

Comparative example A

[0085] Solid materials (F) were dosed continuously to a Lödige CB100 Recycler operating at 1500 rpm. An 18% pre-neutralised LAS acid/LAS mixture (G) was produced separately in an in-line mixing system and pumped into the CB100 mixer at 90°C. Simultaneously with mixture (G), a mixture of nonionic surfactant and fatty acid (H) was pumped into the CB100 mixer at 40°C. The product exiting the CB100 mixer was fed into a Lödige KM10000 ploughshare mixer operating at 450 rpm, as well some zeolite MAP (I) for layering the product. The resulting concentrated powder was then cooled to ambient temperature (the temperature of which varied during the process, but was always between 25 and 37°C) in a fluidised bed using dehumidified air at 10°C. Finally, coarse material larger than 2000 microns was removed from the product by sieving. The resulting product was a free-flowing concentrated powder (J) with a bulk density of between 790 and 810 g/l and a mean particle size of between 560 and 680 microns. The total throughput of the process was 18000 kilograms per hour at a power consumption by the CB100 mixer of between 26 and 35 kW. Table 2 lists the weight percentages of the materials that were dosed into the process.

[0086] From Tables 1 and 2, it can be seen that the powders from Example 1 and Comparative Example A have very similar compositions.

Table 2: Raw material flows in comparative process

Dosed in CB100 mixer		wt%
Solid components (F)	Zeolite MAP	49.84
	SCMC	0.93
	Sodium carbonate	9.57
	Sodium acetate	5.98
Partially neutralised surfactant mixture (G)	LAS acid	20.13
	50% NaOH	0.90
Surfactant mixture(H)	Nonionic 7EO	9.17
	Fatty acid	1.49
Dosed in KM 10000 mixer		
layering (I)	Zeolite MAP	1.99

Example 2

[0087] The strengths of tablets made by compression of the concentrated base powder (E) from Example 1 and the concentrated base powder (J) from Comparative example A were compared.

[0088] From both powders, the particles smaller than 250 microns and greater than 1000 microns were removed by sieving. The powders were then compressed into tablets with a weight of 40 grams with a Graceby Specac air press at a dwell time of 20 seconds, using a die with a diameter of 44 millimetres in order to obtain tablets with a thickness of 17.9 millimetres.

[0089] The results, given in Table 3, show the tablet strength benefits for the concentrated base powder of the invention over the base powder made via the comparative process.

Table 3: Strength of compressed base powders

	Base powder (E)	Comparative base powder (J)
Fmax (N)	173.4	168.3
DFS (kPa)	138.8	134.5

Example 3

[0090] The dissolution times of various sieved fractions of powders (E) and (J) were measured according to the method described above. The results are given in Table 4 and show that the concentrated base powder of the invention (E) surprisingly has improved dissolution over the apparently almost identical composition base powder produced via the comparative method (J).

Table 4: Dissolution time of concentrated base powders

Powder fraction (microns)	Dissolution time of powder (E) (seconds)	Dissolution time of Comparative powder (J) (seconds)
355-500	62	68
500-710	82	92
710-1000	120	133

Example 4

[0091] After 4 weeks warehouse storage in closed big bags, the concentrated base powder (E) from Example 1 was mixed with other detergent ingredients to make a fully formulated powder (K). The concentrated base powder (J) from

Comparative example A was mixed with identical detergent ingredients to make comparative fully formulated powder (L). All ingredients except the perfume were granular materials. The perfume was sprayed onto the fully formulated powders. Compositions of (K) and (L) are given in Table 5.

Table 5: Fully formulated powders

	Fully formulated powder (K)	Comparative fully formulated powder (L)
Concentrated base powder (E)	52.30	
Concentrated base powder (J)		52.30
Antifoam granules	0.82	0.82
Sodium disilicate	2.50	2.50
Sodium citrate	1.17	1.17
Sodium carbonate dense granular	8.20	8.20
Sodium acetate	31.07	31.07
Citric acid anhydrous	1.00	1.00
Sodium Sulphate	0.45	0.45
Polyvinyl pyrrolidone (PVP K30)	0.27	0.27
Enzymes	0.45	0.45
Phosphonates (Dequest(TM))	1.33	1.33
Perfume	0.44	0.44

[0092] Fully formulated powders (K) and (L) were both compressed into tablets of 41.8 grams with a diameter of 44 millimetres and an F_{max} of 38 Newton using a Fette rotating tabletting press. Powder (K) was converted into tablets coded (M) and powder (L) was converted into comparative tablets coded (N). The tablets were stored in closed plastic buckets at room temperature (25°C) for 1 week, after which F_{max} was determined again. The results for tablets (M) and (N) are given in Table 6.

Table 6: Tablet properties

	Tablets (M)	Comparative Tablets (N)
F_{max} after 1 week (N)	46.55	45.69
Standard deviation F_{max} after 1 week (N)	2.26	1.58

Claims

1. Detergent powder manufacturing process in which solid alkali particulate material is blended and reacted with liquid anionic acid surfactant precursor in a mixer to form anionic surfactant and the solid particulate alkali is also blended with nonionic surfactant added in a liquid form to the same mixer, **characterised in that** the nonionic surfactant is added in admixture with anionic surfactant which has been neutralised using a liquid alkaline solution.

2. Process according to claim 1 in which the solid alkali is blended with the nonionic surfactant liquid before it is blended with the liquid anionic surfactant acid precursor.

3. Process according to claim 2 in which the solid alkaline material is sodium carbonate.

4. Process according to any preceding claim in which the anionic surfactant made by reaction with the solid alkali is the same as that made by reaction with the liquid alkaline solution.

5. Process according to claim 4 in which the anionic surfactant is LAS.

6. A powder detergent formulation including at least 20% of the powder formed according to any proceeding claim in admixture with up to 80% of post dosed granules selected from the list comprising, bleach, bleach activator, builder, soil release agents, coloured speckles, buffer, and other detergent formulation ingredients as defined herein.

7. A tablet or discrete region thereof formed by compression of the powder according to claim 6.

8. Use of the tablet according to claim 7 in laundering of clothes.

9. A granular detergent powder with a bulk density of more than 650 g/l obtained by the continuous process of:

a. Forming agglomerates from a finely dispersed powder comprising an alkali source in a high speed mechanical mixer by the addition of two separate liquid binders, in which:

- a first liquid binder is a neutralised mixture of anionic and nonionic surfactants and 0.1-10% water, and
- a second liquid binder is an acid precursor of an anionic surfactant

b. Densification of said agglomerates in a medium speed mechanical mixer while adding 0.1-10% of a finely dispersed powder, preferably sodium aluminiumsilicate, and
c. Drying and/or cooling said agglomerates in a fluidised bed mixer.

10. A granular detergent powder according to claim 9 in which the first liquid binder comprises up to 10 wt% of soap anionic surfactant.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 06 25 1219

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	23 August 2006	Bertran Nadal, J

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EUROPEAN SEARCH REPORT

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