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(54) Detergent tablet compositions

(57) Detergent tablet comprising at least one phase of compacted material and at least one smooth phase, wherein the detergent tablet comprises at least one functional substance having functionality in the washing process and/or in the detergent tablet, characterised in that the functional substance is included in the smooth phase in such an amount that the appearance of the smooth phase is notably different from a tablet with a smooth phase without the functional substance.

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Description

Field of the invention

[0001] This invention relates to detergent compositions in the form of tablets for example, for use in fabric washing or machine dishwashing.

Background to the invention

[0002] Detergent compositions in tablet form have advantages over powdered products in that they do not require measuring and are thus easier to handle and dispense into the wash-load.

[0003] Tablets comprising two or more separate regions have been described. For example WO 01/42416 describes the production of multi-phase moulded bodies comprising a combination of core moulded bodies and a particulate premix. WO 00/61717 describes a detergent tablet which is characterised in that at least part of its outer surface is semi-solid. WO 00/04129 describes a multi-phase detergent tablet comprising a first phase in the form of a shaped body having at least one mould therein and a second phase in the form of a particulate solid compressed within said mould. Recently it has been suggested, for example in EP 1,371,729, EP 1,405,900, EP 1,382,368, EP 1,375,636, EP 1,405,901, EP 1,405,902, EP 1,418,224 and WO 03/104380 to prepare tablets comprising a smooth or semi-solid phase optionally in combination with a solid phase.

[0004] DE19925503 descibes a detergent tablet comprising at least one phase of compacted material and at least one smooth phase, wherein the detergent tablet comprises at least one functional substance, wherein the functional substance is an optical brightener.

Summary of the invention

[0005] An object of the present invention is to provide detergent tablets which contain ant least one functional substance. Another object is to obtain tablets that may be prepared in a robust production process. Still a further object is to provide such tablets having an improved appearance. One or more of these objects are attained by the invention.

[0006] Accordingly in a first aspect the present invention therefore provides a detergent tablet comprising at least one compressed phase of compacted particulate material and at least one smooth phase, wherein the detergent tablet comprises at least one functional substance having functionality in the washing process and/or in the detergent tablet, characterised in that the functional substance is included in the smooth phase in such an amount that the appearance of the smooth phase is notably different from a tablet with a smooth phase without the functional substance.

[0007] Preferably the functional substance is one or more substances chosen from the group consisting of opacifiers, fluorescers, pearlescents, sequestrants and antifoam agents.

Detailed description of the invention

[0008] Weight percentages (wt%) herein are calculated based upon total weight of the tablet, unless otherwise indicated.

[0009] Preferably tablets of the invention are of cylindrical shape (e.g. round, rectangular or square) wherein the two main surfaces (upper side and bottom side) are substantially flat.

[0010] The detergent tablet according to the invention comprises at least one (first)compressed phase of a compacted particulate material and at least one smooth phase. Optionally other phases may be present.

[0011] The regions or phases of a multi-phase tablet are preferably separate layers within a detergent tablet. However, a discrete region of a tablet could also have other forms for example one or more core(s) or insert(s).

[0012] The phases and the other characteristics of tablets according to the invention will now be described in more detail.

Compressed phase (1st phase)

[0013] The compressed phase is a phase or region formed by compacting particulate starting material. Preferably the compressed phase is a solid phase.

[0014] In laundry tablets, the particulate starting material of the compacted phase has a dual function, i.e. it carries liquid like surfactants such as LAS and nonionic and it acts as a binder to give the compacted phase sufficient hardness to survive supply chain and consumer handling. The starting particulate material is preferably made by granulating detergent ingredients to form a powder (base powder) to which other detergent ingredients may be added after granulation (postdosed).

[0015] Preferably the particulate material has a mean particle size in the range from 200 to 2000 μm, more preferably

from 250 to 1400 μm. Fine particles, smaller than 180 μm or 200 μm may be eliminated by sieving before tableting, if desired, although we have observed that this is not always essential.

[0016] While the starting particulate material may in principle have any bulk density, the present invention is especially relevant to tablets made by compacting powders of relatively high bulk density, because of their greater tendency to exhibit disintegration and dispersion problems. Such tablets have the advantage that, as compared with a tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

[0017] Thus the starting particulate composition may suitably have a bulk density of at least 400 g/litre, preferably at least 500 g/litre, and perhaps at least 600 g/litre. gramsgramsgrams

[0018] Although the compacted phase may comprise surfactant materials, when the tablet comprises also a smooth phase, then the compacted phase preferably comprises substantially no surfactants, but ingredients other than surfactants. Examples of these ingredients are for example builders, bleach system, enzymes etc. Preferably the builders in the tablet are predominantly present in compacted phase. Preferably also the bleach system is predominantly present in the compacted phase. Preferably the enzymes are predominantly present in the compacted phase.

[0019] For the purpose of this invention, unless stated otherwise, the term "predominantly present" refers to a situation wherein at least 90 %wt of an ingredient is present in the third region, more preferred more than 98 %wt, most preferred substantially 100 %wt.

[0020] The compacted phase is preferably present as a distinctive region preferably having a weight of 60-95wt%, more preferably 70-80wt%, most preferably about 77wt% of tablet weight.

Smooth phase (2nd phase)

[0021] According to the invention, the detergent tablet comprises a smooth phase, which comprises a functional substance.

[0022] For the purpose of this invention the term smooth phase refers to compositions which are on the one hand solid enough to retain their shape at ambient temperature and on the other hand smooth in appearance. Smooth textures are generally of low or no porosity and have -at normal viewing distance- the appearance of a continuous phase for example as opposed to porous and particulate appearance of a compacted particulate material.

[0023] A functional substance is herein defined as a substance that has functionality in the tablet or in the wash. Preferably the functional substance has functionality in the wash.

[0024] Preferably the functional substance is one or more substances chosen from the group consisting of opacifiers, fluorescers, pearlescents, water-soluble dyes, sequestrants and antifoam agents.

[0025] A preferred functional substance is a fluorescer (optical brightener), for example, Tinopal (Trade Mark) 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-(phenyl-styryl) disulphonate.

[0026] Another preferred functional substance is an anti-foam agent. An antifoam agent is advantageously included, especially if a detergent 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 up to 5% by weight of the composition. The addition of antifoam agent to the smooth phase has the advantage that the antifoam agent is in that phase of the tablet that usually contains the major part of the surfactants that cause foaming (such as LAS and non-ionics).

[0027] Another preferred functional substance is a pearlescent. Preferred pearlescents are beeswax, triacontanyl palmitate, octadecyl octadecanoate , dodecyl hexadecanoate, cetyl palmitate, lanolin, canauba wax, glycol disteatate and jojaba oil. These materials form tiny flakes that give a pearlescent effect (the tiny flakes reflect the light). When a pearlescent is used, the smooth phase preferably should not contain any ingredients that substantially reduce the transparency or translucency of the smooth phase.

[0028] Another preferred functional substance is an opacifier. An opacifier will change the light diffraction characteristic of the smooth phase. Suitable opacifiers are metal oxides and metal hydroxides, such as aluminium hydroxide, aluminium oxide, antimony oxide, titanium oxide. The colour of the opacifier may be used to give the smooth layer a specific colour. Most preferred opacifier is titanium dioxide (which is white).

[0029] Especially preferred as functional substance is a mixture of opacifier and fluorescer, more preferably titanium dioxide and fluorescer, most preferably about 0.4wt% titanium dioxide and 0.5 wt% CBS X (fluorescer in 33wt% slurry in water). This preferred embodiment makes it possible to prepare a tablet of one color (Smooth layer and compressed layer have the same color, e.g. white) but still with different appearance of the layers. In consumer tests such a tablet was especially attractive to the consumer because of the bright white appearance of the smooth layer. Additionally, the color could be used to identify the type of laundry tablet according to the invention (e.g. blue for bleach containing tablets).

[0030] Preferably the functional substance is heat stable. Heat stable herein means that the functional substance is

able to withstand the temperatures used in the preparation of the smooth phase, e.g. about 80°C, for relatively long times, e.g. several days, without substantial decomposition.

[0031] Preferably the functional substance added to the other smooth phase ingredients as a liquid substance. This is advantageous for dispersion of the functional substance in the other ingredients. For this reason functional substances that are available in liquid form are preferred.

[0032] The preferred amounts of the functional substance (based on weight of the smooth phase) is described hereunder in table 1:

Table 1: Preferred functional substances

Functional Substance	Examples	Preferred amount (on smooth phase) (wt.%)	More preferred amount (wt. %)
Opacifier	Titanium dioxide Acusol®	0.01-2	0.1-0.8
Fluorescer	CBS and Tinopal® ex Ciba	0.1-10	1-5
Sequestrant	Phosphonates, e.g. Dequest® ex Solutia	0.1-10	1-5
Antifoam agent	Silicone antifoam agents	0.1-10	1-5
Pearlescent	Lanolin	0.01-2	0.1-0.8
Gas	Air	0.1-10 vol%	1-5 vol%

[0033] Preferably the functional substance is included in a smooth phase which itself is transparent or translucent. Preferably, this means that the composition (without the functional substance) has an optical transmissivity of at least 10%, most preferably 20%, still more preferably 30%, through a path length of 0.5 cm at 25° C. These measurements may be obtained using a Perkin Elmer UV/VIS Spectrometer Lambda 12 or a Brinkman PC801 Colorimeter at a wavelength of 520nm, using water as the 100% standard.

[0034] By the addition of functional substance, the originally transparent starting material may loose its transparency, since the mixture may become a dispersion. Nevertheless, in most cases it may be possible to detect e.g. by microscopy, that despite the addition of the functional substance, in the smooth phase still transparent or translucent regions, i.e. the continuous phase of the dispersion, are present.

[0035] By controlling the addition of the functional substance to the smooth layer, especially the type of the functional substance and the amount added, and optionally the addition of colour, e.g. water-soluble dye, a broad range of different appearances of the smooth layer is attainable according to the invention. For instance it is possible by the addition of opacifiers and water-soluble dye to produce smooth layers with pastel colours.

[0036] Another advantage of the controlled addition of the functional substance is that the process for preparation of the tablets with a smooth phase becomes more robust. In the preparation of the mixture for preparing the smooth phase, the smooth phase ingredients are mixed and heated to relatively high temperature, e.g. to 80°C. Usually the mixture is made in bulk and kept at this high temperature, so that the mixture should be temperature stable for several days. Some thermal breakdown of ingredients in the mixture will lead to slight discoloration of the smooth phase, in particular with transparant smooth phases. Especially when process irregularities occur, often a very light brown or yellow discolouration is observed.

[0037] We have found that by the controlled addition of functional substance according to the invention the discolouration may be overcome in a controlled way, and accordingly the process for preparation of the smooth phase is more robust.

[0038] Other ingredients which may be included in the smooth phase are as follows.

[0039] The smooth region of the tablet may also contain diluent materials for example polyethyleneglycol, dipropyleneglycol, isopropanol or (mono-)propyleneglycol. Preferable the level of these diluents is from 0 to 40 %wt, more preferred 1 to 20, most preferred from 4 to 15 %wt based on the weight of the smooth phase.

[0040] The smooth phase preferably comprises no or only low levels of water. Preferably the level of water is less than 20 wt % based on the weight of the smooth phase, more preferred less than 15 wt%, most preferred from 5 to 12 wt%. Most preferably the smooth phases are substantially free from water, which means that apart from low levels of moisture (e.g. for neutralisation or as crystal water) no additional added water is present.

[0041] In an advantageous embodiment of the invention the smooth phase comprises from 30-100 %wt of non-soap surfactants, more preferred 40 to 90 %wt (based on the total weight of said smooth phase), more preferred from 50 to 80 %wt. It has been found that the combination of a separate smooth first region and these high non-soap surfactant levels provide very good dispersing and cleaning properties to the tablet.

[0042] The smooth region advantageously comprises 50-100 %wt of non-soap surfactants for examples 60 to 90 %wt in combination with optional ingredients such as to 0 to 50 wt% soluble materials (as described above) 0 to 40 wt% diluent materials (as described above) and 0 to 20 wt% (as described above) of water.

[0043] The non-soap surfactants in said smooth phase may for example be anionic, nonionic or cationic non-soap surfactants or mixtures thereof. Relatively low levels of soap may also be present, for example up to 10 %wt based on said third smooth phase.

[0044] Preferably the total weight of surfactants in the smooth phase is from 2 to 20 grams, more preferred from 3 to 10 grams.

[0045] The smooth phase is preferably present as a distinctive region having a weight of 5-30wt.%, more preferably 10-20wt%, most preferably about 16wt% of the tablet weight.

Other phases

[0046] Optionally the tablets according to the invention may comprise other phases then those described above.

[0047] In a preferred embodiment, the tablets comprise a barrier phase which separates the 1st compressed phase and the 2nd smooth phase, which forms a barrier between the 1st and the 2nd phase.

[0048] The barrier phase may be prepared by any suitable method e.g. mixing, casting, spraying etc. In a preferred method said barrier phase is obtained from the compression of particles comprising at least 10wt% of a deformable material. Such particles preferably comprise at least 10 %wt, more preferably at least 50%wt (based on the particles) of the plastically deformable materials.

[0049] For the purpose of the invention the term plastically deformable material refers to any material which on the one hand can exist in a particulate form at ambient temperatures of say 15 to 25°C and preferably somewhat above, but which under moderate pressure of say from 0.1 to 10, especially from 0.2 to 2 kN/cm^2 can merge or flow together when subjected to the compaction pressure.

[0050] Preferably the plastically deformable material has a softening or melting temperature above ambient, conveniently above 35°C, better at least 40°C, Preferably the melting temperature does not exceed 80°C, or even 70°C. Also preferably the plastically deformable materials are water-soluble. A solubility of at least 10g per 100g deionized water at 20°C is desirable. A higher solubility, such as at least 20g per 100 g, is preferred.

[0051] Preferred plastically deformable materials are for example organic polymers containing polar groups, especially polyethylene glycol (PEG). Polyethylene glycols of molecular weight from 1000 to 10,000 and above have been found suitable, especially those of molecular weight in a range from 1000 to 5000. Other plastically deformable organic polymers with a high proportion of hydrophilic groups could be employed. Possibilities are polyacrylates and polyvinyl pyrrolidone. The plastically deformable material may have surfactant properties for example nonionic surfactants containing an average of 20 or more (preferably 30 or more) ethylene oxide residues, anionic surfactants for example particles of linear alkyl benzyl sulphonate and soap particles.

[0052] Preferably the level of plastically deformable material in the barrier phase is from 10 to 90 wt%, more preferred from 20 to 85 wt%, most preferred from 30 to 70 wt% based on the weight of the barrier phase.

[0053] According to a preferred embodiment the starting material of the barrier layer is a high amphiphilic material, such as surfactants, fatty alcohols, fatty acids, fatty amides, or fatty esters. Preferably the amphiphilic materials comprise hydrocarbon chain(s), particularly saturated, of length of 7 carbon atoms or more, preferably 10 carbon atoms or more.

[0054] The barrier phase thus may advantageously be a soap rich phase. Such soap rich barrier phase is hereafter described in more detail.

[0055] For the purpose of the invention the term soap rich phase refers to a separate part of the tablet, e.g. a region, such as a layer, wherein the level of soap is at least 10% wt, more preferably at least 15% wt, still more preferably at least 20% wt, yet more preferably at least 25% wt. Typical soap levels in the soap rich phase are from 15% to 50% by weight, more preferably from 25% to 40% by weight. However, preferably the soap level is at least 50% wt and can even be very high say more than 80wt% or even more than 90 %wt up to 100 %wt based on the weight of the soap rich phase. Other ingredients may also be present in the soap rich layer, although preferably the soap rich layer is substantially free of non-soap surfactants, bleach ingredients and builder materials. Sometimes it may be advantageous however to incorporate into the soap rich layer a highly soluble material such as sugars, urea, alkali metal salts such as sodium chloride etc. Typically such highly soluble materials will have a solubility of at least 100 grams per litre water of 20°C, more preferred at least 250 grams. Advantageously the level of these highly soluble materials in the soap rich phase is less than 50%wt, for example from 5 to 45 %wt, advantageously from 10 to 40 %wt.

[0056] The soap rich region of the tablet may be prepared by any suitable method for example the spraying, applying or brushing of a soap rich formulation, if appropriate followed by hardening e.g. by cooling. In a preferred method the soap rich layer is obtained from the compression of soap rich particles. Such detergent particles preferably comprise at least 10 %wt, more preferably at least 50%wt (based on the particles) of soap surfactants. Suitable detergent particles may for example be granules or other particles having high soap levels, for example soap noodles, marumes or granulates

with high soap levels.

[0057] Preferably the level of soap surfactants in the soap rich particles is more than 50 %wt, more preferred more than 70 %wt, especially preferred from 75 %wt to 100 %wt. Preferably the level of soap rich particles in the soap rich phase is at least 60 wt%, more preferred from 80 to 100 wt%.

[0058] Further surfactants, for example anionic, nonionic or cationic surfactants may equally be present in the soap rich phase for example at a level of 0.1 to 10 %wt based on the weight of the soap rich part. However normally the first soap rich phase will be substantially free from non-soap surfactants.

[0059] In addition to the soap surfactants the soap rich region may comprise other materials for example soluble materials such as electrolyte materials, meltable organic materials and sugars, at a level of 2 to 70 %wt based on the weight of the smooth part, more preferred from 3 to 50 %wt, most preferred 5 to 40 wt%. Examples of preferred materials are water-soluble materials such as the sodium and potassium citrates, sodium chloride, acetates and carbonates, urea and sugar. The water solubility at 20°C of these materials is preferably at least 10 grams per 100 ml of water, more preferred more than 15 grams, most preferably more than 20 grams.

[0060] If these soluble materials are present, their particle size is preferably chosen such that the soap rich phase is a soap rich continuous matrix having dispersed therein particles of the water soluble material.

[0061] It has been found that these materials provide good dissolution properties to the soap rich phase. Furthermore these materials do not negatively affect the desired firm consistency of the soap rich phase.

[0062] The barrier phase is preferably present as a distinctive region preferably having a weight of 1-12wt%, more preferably 3-9wt%, most preferably about 6 wt% of tablet weight.

[0063] The above description of the tablet has been given with reference to a tablet constituted three or four regions. It will however be understood that each of the regions may be composed of a limited number of discrete regions. Similarly the smooth second region or the solid third region may composed of a limited number (say 1-5) of parts e.g. separate layers in the tablet.

[0064] Preferably tablets of the invention are of cylindrical shape (e.g. round, rectangular or square) wherein the two main surfaces (upper side and bottom side) are substantially flat.

[0065] Detergent tablets according to the invention are preferably manufactured by a process involving the application of pressure to a particulate mixture to form the first phase. Advantageously the preparation of the first phase may involve the dosing of a particulate mixture, followed by the exertion of moderate pressure, preferably above the yield stress of the particles.

[0066] A multi-phase tablet comprising a first may advantageously be made by a process, comprising the steps of:

a) a particulate detergent composition is compacted to form a compressed phase;
b) a barrier layer is formed on the compacted phase;
c) a smooth layer is formed on the barrier layer

[0067] In a preferred embodiment of the invention the first particulate composition is pre-compressed at a force of 0.1 to 20 kN/cm^2 between steps (a) and (b). In another preferred embodiment, the particulate composition is flattened between steps (a) and (b).

[0068] Preferably the (co-) compression of the combination of the first and the solid region(s) takes place at a force of from 0.05 to 20 kN/cm^2. Especially if the solid region has been pre-compressed the co-compression in step (c) can advantageously be at a force of 0.1- 10 kN/cm^2, more preferred 0.5 to 5 kN/cm^2. If the solid region has not been pre-compressed, the co-compression preferably takes place at a force of 0.5- 100 kN/cm^2, more preferred 0.7-50 kN/cm^2, most preferred 1-10 kN/cm^2.

[0069] Separately prepared phases can then be adhered to other parts of the tablet for example by gentle pressing or by usage of an adhesive material.

[0070] Similarly a separately prepared solid phase e.g. of compressed particulate materials can be combined with one or more pre-prepared phases e.g. by gentle co-compression.

[0071] A tablet of this invention may be intended for use in machine dishwashing. Such tablets will typically contain salts, such as over 60 wt% of the tablet.

[0072] Water soluble salts typically used in machine dishwashing compositions are phosphates (including condensed phosphates) carbonates and silicates, generally as alkali metal salts. Water soluble alkali metal salts selected from phosphates, carbonates and silicates may provide 60 wt% or more of a dishwashing composition.

[0073] Another preferred possibility is that a tablet of this invention will be intended for fabric washing. In this event the tablet will be likely to contain at least 2 wt%, probably at least 5 wt%, up to 40 or 50 wt% soap surfactant based on the whole tablet, and from 5 to 80 wt% detergency builder, based on the whole tablet.

[0074] Optionally, the tablets according to the invention may be fabric conditioner or fabric softener tablets. Fabric conditioner tablets be based on for instance quaternary ammonium type softeners and/or clays. They are described for instance in WO0181521. In such fabric softener tablets according to the invention preferably the

[0075] Materials which may be used in tablets of this invention will now be discussed in more detail.

Surfactant Compounds

[0076] Compositions which are used in tablets of the invention will contain one or more detergent surfactants. In a fabric washing composition, these preferably provide from 5 to 50% by weight of the overall tablet composition, more preferably from 8 or 9% by weight of the overall composition up to 40% or 50% by weight. Surfactant may be anionic (soap or soap), cationic, zwitter-ionic, amphoteric, nonionic or a combination of these.

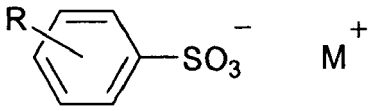
[0077] Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the tablet composition.

[0078] Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkyl-benzene 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.

[0079] Primary alkyl sulphate having the formula

$$ROSO_3^- M^+$$

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 commercially significant anionic surfactant.

[0080] 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.

[0081] In some forms of this invention the amount of non-soap anionic surfactant lies in a range from 5 to 20 wt% of the tablet composition.

[0082] Soaps for use in accordance to the invention are preferably alkali metal or alkaline earth metal salts of naturally occuring 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.

[0083] 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.

[0084] 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.

[0085] 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.

[0086] In some fabric washing tablets of this invention, the amount of nonionic surfactant lies in a range from 4 to 40%, better 4 or 5 to 30% by weight of the whole tablet.

[0087] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition.

[0088] In a machine dishwashing tablet the surfactant may be wholy nonionic, in an amount below 5 wt% of the whole tablet although it is known to include some anionic surfactant and to use up to 10 wt% surfactant in total.

Detergency Builder

[0089] A composition which is used in tablets of the invention will usually contain from 5 to 80%, more usually 15 to 60% by weight 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.

[0090] 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:

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

[0091] These materials contain some bound water (indicated as "xH2O") 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 amply described in the literature. 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 commercially available zeolites A and X, the novel zeolite P described and claimed in EP 384070 (Unilever) and mixtures thereof.

[0092] Conceivably a water-insoluble detergency builder could be a layered sodium silicate as described in US 4664839.

[0093] NaSKS-6 is the trademark for a crystalline layered silicate marketed by Hoechst (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.

[0094] 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.

[0095] Non-phosphorous water-soluble builders 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.

[0096] At least one region (preferably the second region) of a fabric washing tablet 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

[0097] Tablets according to the invention may contain a bleach system in at least one region of a tablet, preferably in the second region. This preferably comprises 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% by weight of the composition.

[0098] Preferred inorganic persalts are sodium perborate monohydrate and 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.

[0099] As indicated above, if a bleach is present and is a water-soluble inorganic peroxygen bleach, the amount may well be from 10% to 25% by weight of the composition.

Other Detergent Ingredients

[0100] The detergent tablets of the invention may also contain (preferably in the second region) one 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 compaction to form a tablet.

[0101] It may also be desirable that a detergent tablet of the invention includes an amount of an alkali metal silicate, particularly sodium ortho-, meta- or disilicate. 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 the particulate material which is compacted into tablets.

[0102] A tablet for fabric washing will generally not contain more than 15 wt% silicate. A tablet for machine dishwashing will often contain more than 20 wt% silicate. Preferably the silicate is present in the second region of the tablet.

[0103] Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the second region) 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.

[0104] Further ingredients which can optionally be used in tablets of the invention, preferably in the second region 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 tripolyphospate with preferably at least 40% of the anhydrous phase I form.

Particle Size and Distribution

[0105] The first soap rich region of the detergent tablet may advantageously be prepared by compacting particles with a high soap content as described above. Preferably these particles have a mean particle size of from 100 to 1000 μm. The second region of a detergent tablet of this invention, is a preferably a matrix of compacted particles.

[0106] Preferably the particulate composition has a mean particle size in the range from 200 to 2000 μm, more preferably from 250 to 1400 μm. Fine particles, smaller than 180 μm or 200 μm may be eliminated by sieving before tableting, if desired, although we have observed that this is not always essential.

[0107] While the starting particulate composition may in principle have any bulk density, the present invention is especially relevant to tablets made by compacting powders of relatively high bulk density, because of their greater tendency to exhibit disintegration and dispersion problems.

[0108] Such tablets have the advantage that, as compared with a tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

[0109] Thus the starting particulate composition may suitably have a bulk density of at least 400 g/litre, preferably at least 500 g/litre, and perhaps at least 600 g/litre.

[0110] Tableting machinery able to carry out the manufacture of tablets of the invention is known, for example suitable tablet presses are available from Fette and from Korch.

[0111] Tableting 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 compaction. In order to carry out the tableting at a temperature which is above ambient, the particulate composition is preferably supplied to the tableting machinery at an elevated temperature. This will of course supply heat to the tableting machinery, but the machinery may be heated in some other way also.

[0112] 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 1050gm/litre up to 1600gm/litre.

Method of manufacture

[0113] Detergent tablets according to the invention are preferably prepared by separately preparing a (pre-)tablet of compacted powdered composition and a smooth (pre-) tablet, followed by an assembly process to prepare the multi-phase tablet.

[0114] Accordingly in a a preferred method to prepare the detergent comprises the steps of

- (a) Preparation of a smooth first tablet;
- (b) Compression of the particulate material to form a second compressed tablet of compacted particulate material;
- (c) Assembling the first tablet and the second tablet.

[0115] Preferably the second tablet is manufactured by inserting a particulate composition into a tablet mould followed by compression of the particulate composition part(s) to form a compressed tablet.

[0116] Preferably the first tablet is manufactured by a casting process. Suitable casting process involve the liquidising of the composition of the smooth phase for example by heating followed by inserting the composition into mold, followed by solidification for example by ageing or cooling.

[0117] Especially preferably the casting involves the heating of the composition of the second tablet filling the composition into a mold and cooling.

[0118] The assembly process for combining the first and the second tablet can be any suitable process for example the co-compression or the adhering of the two phases into one tablet.

[0119] Preferably the diameter of the first tablet is substantially the same as the diameter of the second tablet. For the purpose of the invention "substantially the same diameter" means that the diameter of the first tablet differs less than 5 mm with the diameter of the second tablet more preferably less than 3mm or even less than 1mm.

[0120] This allows for ease of assembly and resulting in a multi-phase detergent tablet wherein each of the phases is present as a separate layer of the tablet. If appropriate adhesive materials may be used to improve the connection between the phases of the multi-phase detergent tablet.

[0121] The present invention will now be explained in more detail by way of the following non-limiting examples.

Examples

Examples 1 to 3 and comparative experiment A

[0122] A detergent powder was made of the following composition:

[0123] A detergent base powder, incorporating organic surfactants, a small percentage of crystalline sodium acetate trihydrate, and zeolite MAP was made using known granulation technology. It had the following composition, shown as parts by weight.

Table 1

Ingredient	Parts by Weight
Sodium linear alkylbenzene sulphonate	20.85
nonionic surfactant (C13-C15 branched fatty alcohol 3EO)	3.07
nonionic surfactant (C13-15 branched fatty alcohol 7EO)	5.98
Soap	1.62
zeolite A24	46.70
Sodium acetate trihydrate	5.92
Sodium carbonate	6.60
sodium carboxymethyl cellulose (SCMC)	0.64
additional moisture and minor ingredients	8.50
Total	100

[0124] The amount of zeolite MAP (zeolite A24) in the table above is the amount which would be present if it was anhydrous. Its accompanying small content of moisture is included as part of the moisture and minor ingredients. The base powder and other ingredients were mixed together as set out in the following table 2.

Table 2

Comp A.	Parts by Weight
Base powder, as above	57.18
Antifoam (1)	2.30
Fluorescer (2)	1.59
Sodium percarbonate	19.23

(continued)

Comp A.	Parts by Weight
TAED (3)	6.48
Sodium disilicate	4.07
Soil release polymer (4)	1.40
Acrylic/maleic copolymer (5)	1.53
EDTM phosphonate 0.95 Colored speckles	1.77
Cellulosic swelling disintegrant (6)	3.50
Total	100.0
Wherein: (1) Antifoam is 17%wt silicon oil, 71%wt sodium carbonate and the remainder petroleum jelly and phosphate ester. (2) Fluorescer is 9.9%wt Fluorescer and 82.5%wt sodium carbonate the remainder being minor ingredients (3) TAED is 83%wt TAED in 9%wt sodium sulphate the remainder being minor ingredients. (4) Soil release polymer is 18%wt soil-release polymer, 44%wt zeolite MAP, 21%wt sodium carbonate and minor ingredients.	

(5) Acrylic/maleic polymer is Sokolan CP5 ex BASE (91%wt active) (6) Cellulosic swelling disintegrant (Arbocel ex Rettenmaier)

[0125] The solid phase was made by mixing the ingredients (except for the cellulosic swelling disintegrant) and finally adding the cellulosic swelling disintegrant.

[0126] The solid phase was made in a labscale Graseby Specac tabletting press; and had a strength (expressed in terms of the diametrical fracture stress) of approximately 25k Pa. The diametrical stress is defined as DFS=2*Fmax/(Π*D*t) with Fmax being the tablet strength expressed as the force required to break a tablet when it is applied on its circumference. D the tablet diameter and t its thickness.

[0127] A second exemplary solid phase formulation is exactly as for the above formulation an except that the level of the cellulosic swelling disintegrant is to 1.75% wt (and the base powder increased to 58.93% wt of the final formulation.

[0128] Smooth or semi-solid parts were prepared of the following composition:

Table 3

Ingredient	Composition Parts by wt
Na-las	34
Nonionic(Lutensol AO5)	26
Soap(Pristerene4916)*	5
Mono propylene glycol solvent	27
Water	8
Notes: * is a C16-C18 soap	

Preparation of tablets of examples 1 to 3 and comparative experiment A.

[0129] The smooth phase composition of table 3 was heated to 80°C and the functional substance (1,2,3) was added. In comparative experiment A no functional substance was added (see table 4 for details).

Table 4

No	Functional substance	Amount in gel (smooth phase) (wt. %)	Appearance of gel
1	TiO_2 , and Fluorescer CBS X ex Ciba (33% water slurry)	0.4 0.5	Non-transparent Bright white
2	TiO_2	0.1	Semi-transparent With slight brownish shade
3	TiO_2 Fluorescer as Ex.1 Blue dye (BlueHP ex Milliken)	0.1 0.25 0.025	Semi-transparent Bright blue pastel color
A	No functional substance	-	Transparent Brownish shade

[0130] Subsequently the smooth phase composition was cast into moulds and cooled to 20°C to form firm, 5 grammes smooth and semi-solid parts of 32mm diameter and 6mm high.

[0131] Multi-layer tablets were made by pre-compressing 25 grammes of the powdered composition in a die of 45 mm diameter at 4 kN/cm^2, followed by adherence of a smooth and semi-solid part on top of the compressed layer followed by a final compression step at 0.1 kN/cm^2. During the final compression step, the smooth and semi-solid part flows by the compaction forces to form a 3.4 mm smooth semi-solid layer adhered on top of the compressed particulate layer.

Claims

1. Detergent tablet comprising at least one compressed phase of compacted particulate material and at least one smooth phase, wherein the detergent tablet comprises at least one functional substance having functionality in the washing process and/or in the detergent tablet, **characterised in that** the functional substance is included in the smooth phase in such an amount that the appearance of the smooth phase is notably different from a tablet with a smooth phase without the functional substance.

2. Detergent tablet according to claim 1, wherein tablet is a laundry tablet and the functional substance has functionality in the wash.

3. Detergent tablet according to claim 1 or 2, wherein the functional substance is one or more substances chosen from the group consisting of opacifiers, fluorescers, pearlescents, sequestrants and antifoam agents.

4. Detergent tablet according to any of claims 1-3, wherein the functional substance is a substance which influences the tranparency of the smooth phase.

5. Detergent tablet according to any of claims 1-4, wherein the amount of functional substance is 0.01-10 wt.%, preferably 0.1-5 wt.% based on the weight of the smooth phase.

6. Detergent tablet according to any of claims 1-5, wherein the functional substance is opacifier and the amount of opacifier is 0.01-2 wt.%, preferably 0.1-0.8 wt.% based on the weight of the smooth phase.

7. Detergent tablet according to any of claims 1-6, wherein the functional substance is fluorescer and the amount of fluorescer is 0.1-10 wt.%, preferably 1-5 wt.% based on the weight of the smooth phase.

8. Detergent tablet according to any of claims 1-7, wherein the functional substance is pearlescer and the amount of pearlescer is 0.01-2 wt.%, preferably 0.1-0.8 wt.% based on the weight of the smooth phase.

9. Detergent tablet according to any of claims 1-8, wherein the functional substance is sequestrant and the amount of sequestrant is 0.1-10 wt.%, preferably 1-5 wt.% based on the weight of the smooth phase.

10. Detergent tablet according to any of claims 1-9, wherein the functional substance is antifoam agent and the amount

of antifoam agent is 0.1-10 wt.%, preferably 1-5 wt.% based on the weight of the smooth phase.

11. Detergent tablet according to any of claims 1-10, wherein the functional substance is air and the amount of air is 0.1-10, preferably 1-5 volume percent of the volume of the smooth phase.

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EUROPEAN SEARCH REPORT

Application Number
EP 05 07 6685

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The present search report has been drawn up for all claims

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