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(54) **Detergent compositions**

(57) Detergent tablet having a plurality of discrete phases with different composition, comprising a compressed phase and an encapsulate phase comprising encapsulates, characterised in that the encapsulate

phase is separated from the compressed phase by a barrier phase and process for preparing such tablet.

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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. For the purpose of this invention detergent tablets include tablets suitable for fabric treatment (e.g. fabric softening).

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] A problem with perfume incorporation into detergent tablets is that often perfume ingredients may suffer from a negative interaction with other components. WO00/11132 describes the incorporation of perfume in detergent tablets whereby the perfume is not contained in the same phase with the bleaching agent. However such systems may suffer from bleeding whereby perfume ingredients from one phase of the tablet leak into the other phase. This may still lead to undesirable interaction possibly leading to undesired degradation of ingredients or discoloration of the tablet.

[0005] WO 99/27069 describes detergent tablets comprising a compressed portion and an non-compressed portion wherein the non-compressed portion comprises a perfume component which could be an encapsulated perfume. However the preparation of a non-compressed phase often involves conditions e.g. elevated temperatures which may lead to damage of the encapsulated perfume.

Summary of the invention

[0006] It is an object of the present invention to provide a method to produce a detergent tablet comprising at least two phases, wherein if said tablet comprises perfume, whereby undesired interaction of perfume ingredients with other components can be minimised. Another object of the invention is to try and minimise leakage of perfume out of the detergent tablet thereby maximising the amount of perfume that can be deposited onto the articles to be washed.

[0007] Surprisingly it has been found that advantageously perfume, or any other functional material, can be used in the form of encapsulates, whereby the encapsulates are incorporated into a separate phase.

[0008] According to a first preferred embodiment of the present invention there is provided a detergent tablet having a plurality of discrete phases with different composition, comprising a compressed phase and an encapsulate phase comprising encapsulates, characterised in that the encapsulate phase is separated from the compressed phase by a barrier phase. The separation results in improved integrity of the compacted phase. Also, in a layered tablet, if the encapsulate phase layer is protected on one side by the barrier phase and on the other side by another tablet phase, e.g. a smooth phase, it is possible to achieve a slower release of the perfume encapsulates, which is advantageous, since then more perfume will be released on the washed clothes.

Detailed description of the invention

[0009] Weight percentages (wt% or %wt) herein are calculated based upon total weight of the tablet, unless otherwise indicated.

[0010] The tablets according to the invention comprise at least three phases or regions, a first compressed phase of compacted particulate material, a second barrier phase, a third encapsulate phase comprising the perfume encapsulate and optionally a fourth smooth phase. These phases and the other characteristics of the tablets according to the invention will now be described in more detail.

Compressed phase (1st phase)

[0011] The compressed phase is a phase or region formed by compacting particulate starting material. Preferably the

compressed phase is a solid phase.

[0012] 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).

[0013] 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.

[0014] 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 the greater tendency of high bulk density tablets to exhibit disintegration and dispersion problems. Powders with a high bulk density may be advantageously used to prepare tablets according to the invention and 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.

[0015] 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.

[0016] 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.

[0017] 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.

[0018] 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.

Barrier phase (2nd phase)

[0019] The function of the barrier phase is to provide a phase on which the encapsulate phase can be applied. Preferably the barrier phase is suitable for application of an aqueous encapsulate dispersion. The barrier phase is therefore preferably more hydrophobic than the compacted 1st phase. Another function of the barrier layer may be to form a barrier for water-soluble ingredients between the compacted 1st phase and the smooth 4th phase.

[0020] Preferably, the barrier phase substantially consists of plastically deformable hydrophobic material.

[0021] 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 50wt% (based on the particles) of the plastically deformable materials.

[0022] 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/cm² can merge or flow together when subjected to the compaction pressure.

[0023] 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.

[0024] 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.

[0025] According to a preferred embodiment the barrier layer is an 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.

[0026] Plastically deformable materials may further comprise 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.

[0027] The barrier phase thus may advantageously be a soap rich phase. Such soap rich barrier phase is hereafter described in more detail.

[0028] 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.

[0029] The soap rich region of the tablet may be prepared by any suitable method for example 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.

[0030] 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%.

[0031] 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.

[0032] 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.

[0033] 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.

[0034] 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.

[0035] 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.

Encapsulate phase (3rd phase)

[0036] The encapsulate phase of the tablet comprises encapsulates. The encapsulates contain a functional ingredient, preferably perfume or a laundry finishing agent. Most preferred functional ingredients are perfumes.

[0037] Encapsulates are preferably of the core in shell type such as for example described in GB 0751600, EP 385,534, US 3,341,466 and which are commercially available. Preferred encapsulates are melamine-formaldehyde-urea capsules for example as described in US 6,224,795, US 3,516,941 and US 5,154,842. Encapsulated perfumes may be added as such to the encapsulate phase, but may also advantageously be used e.g. as slurries (preferably as aqueous dispersions). Advantageously the level of functional ingredient (e.g. perfume) in the encapsulate phase is from 1 to 99wt% based on the weight of said phase, more preferred from 10-90wt%, even more preferred 50-90wt%.

[0038] The encapsulate phase is formed by applying an encapsulate containing composition to the barrier phase by any known method. Preferably the phases are layers.

[0039] In a preferred embodiment an encapsulate layer may be formed by applying an aqueous dispersion of encapsulates to the barrier layer. A preferred aqueous dispersion is a core in shell perfume encapsulate of melamine-formaldehyde-urea capsules. In this preferred embodiment, the aqueous dispersion may be used as an adhesive to attach the barrier layer to another layer of the tablet. The aqueous dispersion may be used as adhesive alone or in combination with other adhesives, e.g. in different adhesive layers. Suitable layers which in these way could be attached include but are not limited to a second compressed phase, an extruded phase or a smooth phase. In a particularly preferred embodiment, the aqueous dispersion is used as an adhesive to attach the barrier layer to the smooth layer. The resin (melamine-formaldehyde-urea) which is part of the aqueous dispersion will, when the water has been removed, in part

or in whole, e.g. by evaporation or migration to other parts of the tablet, form a tight bond between the barrier layer and the smooth layer, so that the smooth layer is very effectively attached to the other phases of the tablet. There is no need for applying other adhesives in such case. Notwithstanding the above preferred embodiments, the encapsulates may also be included in addition to any other glue that may be used as adhesive in the attachment of different layers of the tablets according to the invention, e.g. thermoplastic glues. In such case, care should be taken that the integrity of the encapsulates is not harmed, e.g. by avoiding the use of too high temperatures and/or pressures.

[0040] The method of application of the encapsulate composition may be any suitable method, such as for instance spraying, dropping, pouring, casting or extrusion.

[0041] Preferably after application of the encapsulate composition on the barrier phase, the encapsulate composition is cured and/or dried. Drying is especially preferred, when the encapsulate composition is an aqueous dispersion of encapsulates. Drying may be accomplished in any known way e.g. by increasing temperature, applying a gas flow (e.g. air or nitrogen) or by reducing pressure. For instance, in a preferred embodiment a heated aqueous encapsulate dispersion is sprayed or poured on the barrier phase, such that evaporation of water from the composition is promoted.

[0042] The encapsulate phase is preferably present as a distinctive region having a weight of 0.01-10wt.%, more preferably 0.15-5wt.%, even more preferably 0.15-1.5wt.%, most preferably about 0.6wt% of the tablet weight. Generally, more than about 0.15wt.% encapsulate phase is needed for obtaining a glueing effect.

Smooth phase (4th phase)

[0043] In a preferred embodiment of the invention the detergent tablet comprises in addition to the phases described above a fourth phase, which is a smooth phase.

[0044] 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.

[0045] The smooth region of the tablet may also contain diluent materials for example polyethyleneglycol, dipropylene glycol, 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.

[0046] 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.

[0047] Preferably the smooth phase is transparent or translucent. Preferably, this means that the composition 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.

[0048] The transparency or translucency of the compositions according to the invention does not preclude the composition being coloured, e.g. by addition of a dye, provided that it does not detract substantially from clarity.

[0049] 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.

[0050] 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.

[0051] 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.

[0052] Preferably the total weight of surfactants in the smooth phase is from 2 to 20 grams, more preferred from 3 to 10 grams.

[0053] 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.

[0054] The regions or phases of a multi-phase tablet according to the invention are preferably separate layers within the detergent tablet. However, a discrete region of a tablet could also have other forms for example one or more core (s) or insert(s).

[0055] In a preferable embodiment of the invention the barrier phase may comprise a mould, into which the encapsulate phase may be filled or may protrude into.

[0056] Preferably the total weight of the detergent tablet according to the invention is from 10 to 100 grams, more

preferred from 15 to 60 grams, most preferred from 15 to 50 grams.

[0057] In a most preferred embodiment the first phase is a layer of compacted particulate material, on which the barrier phase being a layer of material which plastically deformable under pressure, is applied by pressing; Then the encapsulate phase is applied as a layer on the barrier layer, e.g. by spraying and a fourth smooth layer is applied on the encapsulate layer by gentle pressing.

[0058] 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.

[0059] 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.

[0060] 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.

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 compacted phase;
- b) a barrier layer is formed on the compacted phase;
- c) an encapsulate layer comprising encapsulates is formed on the barrier layer.

[0061] Preferably the first particulate composition is such that upon compression a solid phase of compressed particulate material is formed.

[0062] In a preferred embodiment of the invention the first particulate composition is pre-compressed at a force of 0.1 to 20 kN/cm² between steps (a) and (b). In another preferred embodiment, the particulate composition is flattened between steps (a) and (b).

[0063] Preferably the (co-) compression of the combination of the smooth and the solid region(s) takes place at a force of from 0.05 to 20 kN/cm². 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/cm², more preferred 0.5 to 5 kN/cm². If the solid region has not been pre-compressed, the co-compression preferably takes place at a force of 0.5- 100 kN/cm², more preferred 0.7-50 kN/cm², most preferred 1-10 kN/cm².

[0064] The smooth phase may advantageously also be prepared e.g. by extrusion, spraying, dropping, pouring, casting or other shaping methods.

[0065] 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.

[0066] 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.

[0067] 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.

[0068] 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.

[0069] 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.

[0070] 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 the encapsulate phase should be separated from the compressed phase by a barrier phase.

[0071] Materials which may be used in tablets of this invention will now be discussed in more detail.

Surfactant Compounds

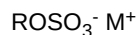
[0072] Compositions which are used in tablets of the invention will contain one or more detergent surfactants. In a fabric washing composition, these preferably provide from 5 to 50% by weight of the overall tablet composition, more preferably from 8 or 9% by weight of the overall composition up to 40% or 50% by weight. Surfactant may be anionic (soap or soap), cationic, zwitter-ionic, amphoteric, nonionic or a combination of these.

[0073] Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to

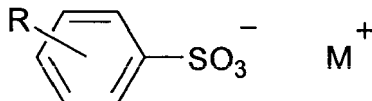
30% or 40% by weight of the tablet composition.

[0074] 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 C₈-C₁₅; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

[0075] Primary alkyl sulphate having the formula



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.

[0076] 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.

[0077] 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.

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 C₁₀ to C₂₀ soaps for example from C₁₆ to C₁₈ or C₁₂ soaps.

[0078] 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.

[0079] Specific nonionic surfactant compounds are alkyl (C₈₋₂₂) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C₈₋₂₀ 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.

[0080] Especially preferred are the primary and secondary alcohol ethoxylates, especially the C₉₋₁₁ and C₁₂₋₁₅ primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

[0081] 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.

[0082] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition, preferably those of the compacted first phase starting particles.

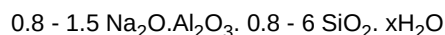
[0083] In a machine dishwashing tablet the surfactant may be wholly nonionic, in an amount below 5 wt% of the whole tablet although it is known to include some anionic surfactant and to use up to 10 wt% surfactant in total.

[0084] In a fabric softening tablet double tailed surfactants or a combination of positively charged molecules and anionic surfactant(s) such as soap may be employed. In addition or otherwise silicone oil may be used as softener.

Detergency Builder

[0085] 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.

[0086] 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:



[0087] 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 SiO₂ 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.

[0088] Conceivably a water-insoluble detergency builder could be a layered sodium silicate as described in US 4664839.

NaSKS-6 is the trademark for a crystalline layered silicate marketed by Hoechst (commonly abbreviated as "SKS-6"). NaSKS-6 has the delta-Na₂SiO₅ 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}·yH₂O 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.

[0089] 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.

[0090] 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.

[0091] At least one region (preferably the compacted 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

[0092] 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.

[0093] 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, tetraacetyethylene 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.

[0094] 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

[0095] The detergent tablets of the invention may also contain (preferably in the compacted 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.

[0096] The detergent tablets of the invention may also contain (preferably in the compacted region) 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.

[0097] An antifoam material is advantageously included (preferably in the compacted region), 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.

[0098] 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.

[0099] 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 compacted region of the tablet.

[0100] Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the compacted 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.

[0101] Preferably the tablet according to the invention comprises a disintegrant, preferably a swellable disintegrant. A disintegrant is known in the art to aid disintegration and quick dissolution of the product in a bucket or bowl of 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). If the tablet contains disintegrant, the disintegrant level is preferably less than 0.5wt% in the barrier layer and more than 0.5wt% in the first compacted phase. Most preferably the barrier layer has no disintegrant and the compacted phase contains all of it.

[0102] Further ingredients which can optionally be used in tablets of the invention, preferably in the compacted 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 tripolyphosphate with preferably at least 40% of the anhydrous phase I form.

[0103] 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 Korsch.

[0104] 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.

[0105] 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.

[0106] The present invention will now be explained in more detail by way of the following non-limiting examples.

Examples

Methods

Tablet Strength

[0107] The strength of the tablets (tablet strength), in their dry state as made on the compaction press, can be determined according to their diametrical fracture stress DFS, which is calculated from the equation:

$$DFS = \frac{2 F_{\max}}{\pi D t}$$

where DFS is the diametrical fracture stress in Pascals, $F_{\max}$ is the applied load in Newtons to cause fracture, D is the tablet diameter in metres and t is the tablet thickness in metres. The test is carried out using an Instron type universal

testing instrument to apply compressive force on a tablet diameter (i.e. perpendicular to the axis of a cylindrical tablet). It is preferred that tablets have a DFS of at least 20 kPa more preferably at least 25 kPa, such as 30 kPa or above.

Example 1

i) Preparation of a detergent starting powder suitable for compaction:

[0108] A powder was made of the following composition by pre-granulating the granule ingredients, followed by post-dosing the rest of the ingredients.

Table 1

Compaction device: tableting machine Batch size: 75 kg	
<i>Base Powder Ingredient</i>	<i>Parts by weight</i>
Linear alkylbenzene sulphonate, sodium salt	8.5
Alcohol ethoxylate nonionic, avg. 7EO	3.7
Zeolite A24	19.0
Sodium salts	5.2
Sodium carboxymethyl cellulose	0.4
Soap C16-C18	0.7
Moisture	3.4
<i>Post dose ingredient</i>	
Antifoam adjunct	0.6
Fluorescor adjunct	1.5
Tetraacetyldiethylenediamine (TAED)	2.9
	10.9
Sodium percarbonate	1.3
Sesquestrant	2.2
Sodium di-silicate	38.1
Sodium tripolyphosphate Minors (citric acid, enzymes)	to 100

ii) Preparation of a compacted phase

[0109] Preparation of a first solid compacted phase:

24 g of the particulate composition are inserted into a 45 mm die of a tableting machine, optionally followed by a flattening step. The whole material is compressed at 34 kN into a single tablet with diameter 45mm.

iii) Preparation of two-layer detergent tablets (compacted phase and barrier phase):

[0110] 1 part by weight of C12-C14 soap particles was mixed with 1 weight part of sodium sulphate. 2 g of the mixture were dosed in the tablet mould on top of the tablet prepared in step ii), followed by a second compression step of 28 kN.

iv) Preparation of perfume-capsules coated tablets

[0111] 400 mg of slurry containing perfume capsules (commercially available melamine-formaldehyde-urea capsules) was applied drop-wise on top of tablets made in iii).

[0112] After formation of the tablets the extent to which perfume microcapsules were broken was determined by checking leakage of perfume from the tablet by dissolving the tablet in water at 40°C and measuring the perfume headspace above the solution after 30 min. The higher the perfume concentration in the headspace, the greater the number of capsules that were broken during tableting. The leakage was calculated as a % of what the leakage would have been had all the microcapsules suffered damage.

The tablet strength was measured resulting in a tablet with a strength of 59 N. The results are given in table 2.

Comparative experiment APreparation of perfume-capsules coated tablets

[0113] On a compacted phase tablet as prepared in example 1, step ii) an encapsulate phase was applied. 400 mg of slurry containing perfume capsules (commercially available melamine-formaldehyde-urea capsules) was applied drop-wise on top of the tablets made in example 1, step ii) but the tablet no longer had a smooth surface because of partial dissolution/swelling of tablet ingredients. Moreover the tablet lost strength with time. Comparative experiment A shows that applying a perfume encapsulate slurry on the first compacted phase is not feasible.

Comparative experiment Bi) Preparation of first compacted phase with perfume capsules:

[0114] A concrete mixer is fed with detergent powder having the formulation given in the table above, onto which a slurry of perfume capsules (melamine-formaldehyde-urea capsules incorporating a perfume which is commercially available) composition is applied by spraying with a nozzle.

[0115] Spraying system: spray nozzle for STREA-1 NIRO

[0116] Nozzle spec: 1.1 mm

[0117] Mixer: standard concrete mixer

[0118] Spray-on rate: 0.6 kg hr⁻¹

[0119] Capsule concentration in slurry: 42wt% (balance: water) Batch size: 75 Kg

[0120] Compaction device: tableting machine

[0121] 24 g of the particulate composition are inserted into a 45 mm die of a tableting machine, optionally followed by a flattening step. The whole material was compressed at 32 kN into a single tablet.

ii) Preparation of two-layer detergent tablets (compacted phase and barrier phase):

[0122] Two-layer tablets were prepared as in example 1, step iii) with compaction force of second phase of 27kN. The tablet strenght was 34N.

Comparative experiment C

[0123] A compacted phase and a two-layer tablet was prepared as in comparative experiment B, steps i) and ii), but with different compression pressures indicated in table 2. The tablet strength was 60 kN.

[0124] The tablet strength and leakage were measured according to the methods of example 1. Results are given in table 2.

Table 2: Leakage results:

Example	Compaction force first phase (kN)	Compaction force second phase (kN)	Tablet strength (N)	Leakage (%)
1	34	28	59	1
B	32	27	34	73
C	53	30	60	81

[0125] Clearly, in the tablets of invention (example 1) where the perfume capsule dispersion was applied on the barrier layer (less soluble solid phase) much less breakage of capsules occurs than in comparative experiments B and C.

[0126] To determine the solubility in water at 20°C of a compressed particulate phase (in table designated as: white and barrier phase, samples were prepared as in table 3 by pressing tablet ingredients as in example 1. Solubility was measured using a T90 dissolution method. T90: Is the time in minutes when 90 % of a single tablet is solved in 9 liter water (20°C ± 0.5) with the help of a conductance measuring equipment. Results are described in table 3.

Table 3: Tablet dissolution rate (T90 values)

Sample	Thickness (mm)	Diameter (mm)	Weight (g)	Force @ Failure N	DFS kPa	T90 (min.)
Barrier/25g/60N	10.95	44.10	24.92	65.27	86.06	23.3
Barrier/25g/40N	10.89	44.58	24.06	47.95	62.87	22.4
Barrier/20g/60N	8.84	45.76	20.11	56.37	88.72	20.7
Barrier/20g/40N	8.93	47.00	19.73	36.51	55.35	21.0
White/25g/60N	12.25	45.92	24.84	59.24	67.14	1.8
White/25g/40N	12.54	45.31	24.81	42.02	47.13	1.6
White/20g/60N	9.97	45.33	20.10	63.05	88.84	2.2
White/20g/40N	10.27	46.44	20.03	39.89	53.32	1.8
Tablet Ex. 1	11.87	45.67	26.15	59.10	39.24	3.6

[0127] From table 3 it is seen that for a comparable strength of the layers, e.g. Barrier/20g/40N and White/20g/40N, the T90 of barrier layer is far higher (about 10 times) as compared to T90 of the compacted white layer: Hence this shows the barrier layer dissolves slower as compared to compressed phase (white) layer.

Claims

1. Detergent tablet having a plurality of discrete phases with different composition, comprising a compressed phase and an encapsulate phase comprising encapsulates, **characterised in that** the encapsulate phase is separated from the compressed phase by a barrier phase.
2. Detergent tablet according to claim 1, wherein the barrier phase is less soluble in water at 20°C than the compressed phase.
3. Detergent tablet according to claim 1 or 2, wherein the detergent tablet additionally comprises a smooth phase.
4. Detergent tablet according to claim 3, wherein the encapsulate phase is located between the smooth phase and the barrier phase.
5. Detergent tablet according to any of claims 1-4, wherein the phases of the tablet are layers of the tablet.
6. Detergent tablet according to any of claims 1-5 wherein the encapsulates are core in shell encapsulates.
7. Detergent tablet according to any of claims 1-6, wherein the encapsulates comprise perfume.
8. Detergent tablet according to any of claims 1-7, wherein the encapsulates comprise finishing agent.
9. Detergent tablet according to any of claims 1-8, wherein the encapsulates are melamine-formaldehyde-urea encapsulates.
10. Detergent tablet according to any of claims 1-9, wherein the tablet comprises a disintegrant, wherein the disintegrant is substantially absent in the barrier phase.
11. Detergent tablet according to any of claims 1-10, wherein the encapsulate phase is used as glue, alone or in

combination with other glue between at least two tablet phases.

- 12.** Process for the preparation of a detergent tablet having a plurality of discrete phases with different composition, wherein:

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- a) a particulate detergent composition is compacted to form a compacted phase;
- b) a barrier layer is formed on the compacted phase;
- c) an encapsulate layer comprising encapsulates is formed on the barrier layer.

- 13.** Process according to claim 11, wherein the encapsulate layer is formed by applying on the barrier layer an aqueous suspension of the encapsulates.

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- 14.** Process according to claim 11 or 12, wherein the encapsulates are core in shell encapsulates.

- 15.** Process according to any of claims 11 to 13, wherein the encapsulates are melamine-formaldehyde-urea encapsulates.

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- 16.** Process according to any of claims 11 or 14, wherein a smooth layer is formed on the encapsulate layer.

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European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 05 07 6686

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Place of search The Hague		Date of completion of the search 6 January 2006	Examiner Bertran Nadal, J
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**ANNEX TO THE EUROPEAN SEARCH REPORT
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