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(54) **Use of detergent tablets**

Verwendung von Waschmitteltabletten

Utilisation des comprimés détergents

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(73) Proprietors:
• **UNILEVER N.V.**
3013 AL Rotterdam (NL)
Designated Contracting States:
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• **UNILEVER PLC**
London EC4P 4BQ (GB)
Designated Contracting States:
CY GB IE

(72) Inventors:
• **van der Kooij, Felix Marco**
3133 AT Vlaardingen (NL)
• **Verschelling, Gilbert Martin**
3133 AT Vlaardingen (NL)

(74) Representative: **Joppe, Hermina Laura Petronella
et al**
Unilever Patent Group
Olivier van Noortlaan 120
3133 AT Vlaardingen (NL)

(56) References cited:
EP-A- 1 371 721 WO-A-00/61717
US-B1- 6 551 981

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Description**FIELD OF THE INVENTION**

[0001] This invention relates to use of a cleaning composition in the form of a tablet for use in fabric washing.

BACKGROUND OF 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 washload.

[0003] Single phase tablets of a cleaning composition are generally made by compressing or compacting a quantity of the composition in particulate form.

[0004] Tablets comprising two or more separate regions have also been described. For example WO-A-01/42416 describes the production of multi-phase moulded bodies comprising a combination of core moulded bodies and a particulate premix. WO-A-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. WO-A-00/61717 describes a detergent tablet which is characterised in that at least part of its outer surface is semi-solid. The latter disclosure states that the semi-solid layer can be rubbed onto heavily stained areas of the fabric to pretreat them, thereby assisting removal of the stains in the wash. This may be regarded as an extension of the concept of rubbing stains with a pretreatment composition, e.g. in the form of a stick or bar. There is already a host of references in the art, relating to such pre-treatment products, for example US-A-4 396 521 and EP-A-205 999.

[0005] Whilst in principle, this method of pre-treatment with a two phase tablet appears to be workable, in practice, it is desirable for the semi-solid phase to contain a relatively high level of liquid component(s), especially organic solvent. This results in a problem that liquid can bleed into the compressed particulate solid layer leading to a degradation in tablet appearance and/or integrity.

[0006] We have now found that the aforementioned problem can be mitigated or overcome by having an intermediate layer between the semi-solid layer and the compressed particulate layer. However, since the regions of different material may in the alternative be arranged in other than layered fashion, the more generic term "phase" is used in many places throughout this specification.

DEFINITION OF THE INVENTION

[0007] According to a first aspect of the present invention, there is provided use of a multi-phase laundry cleaning tablet comprising a compressed particulate phase and a semi-solid phase for pretreating a region of a fabric prior to washing by rubbing the semi-solid phase on the region, wherein an intermediate phase is at least partly situated between the semi-solid phase and the compressed particulate phase, and wherein the intermediate phase comprises at least 10% by weight of soap.

[0008] A second aspect of the present invention provides a method of pretreating a region of a fabric prior to washing by rubbing the region with a semi-solid phase of a multi-phase laundry cleaning tablet comprising the semi-solid phase and a compressed particulate phase, wherein an intermediate phase is at least partially situated between the semi-solid phase and the compressed particulate phase and wherein the intermediate phase comprises at least 10% by weight of soap.

[0009] A third aspect of the present invention provides a method of washing a fabric, comprising washing the fabric in a wash liquor in which a multi-phase laundry cleaning tablet has been dispersed and/or dissolved, a region of the fabric having first been rubbed with a semi-solid phase of the tablet, wherein the tablet further comprises a compressed particulate phase and wherein an intermediate phase is at least partially situated between the semi-solid phase and the compressed particulate phase and wherein the intermediate phase comprises at least 10% by weight of soap.

DETAILED DESCRIPTION OF THE INVENTION

[0010] As used herein, the term "phase" refers to a region or a plurality of regions of a tablet having a particular composition and/or physical form such as solid homogeneous mass, compressed particulate mass or semi-solid such as gel. These regions may be arranged as layers or as islands at least partly surrounded by one or more other such regions.

[0011] The tablets utilised in the present invention may be any suitable shape, e.g. spherical or polyhedral. However, preferably they are of cylindrical shape wherein the two main surfaces (upper side and bottom side) are substantially flat. In another preferred class of embodiments, the surface of the semi-solid layer is dome-shaped to facilitate pre-treatment application.

[0012] The multi-phase tablets used in the present invention comprise a semi-solid phase, an intermediate phase and

additionally one phase of compacted particulates. Suitably, there may be further phases.

[0013] The intermediate phase is at least partly situated between the semi-solid phase and the compressed particulate phase. That is to say, at least part of the area where the semi-solid phase would be in contact with the compressed particulate phase at an interface therebetween, an intermediate phase intervenes. However, some semi-solid phase could still be in direct contact with compressed particulate phase. Most preferably though, substantially no semi-solid phase is in direct contact with compressed particulate phase. The intermediate phase itself may actually comprise regions of intermediate phase of respective composition and/or physical state which are different from each other. The only requirement is that they each differ from the semi-solid phase and the compressed particulate phase. In the case where the compressed particulate phase and/or semi-solid phase are in the form of a plurality of discrete islands, two or more such islands could be respectively isolated at the interface by a different intermediate phase.

[0014] One or more other phases than compressed particulate, semi-solid and intermediate may also be present in contact with any one or more of the latter phases.

[0015] Most advantageous though, is the location of the intermediate phase substantially complete between the compressed particulate phase and the semi-solid phase in a stackered layer structure.

The Intermediate Phase

[0016] The intermediate phase is intended to reduce bleeding of liquid from the semi-solid phase into the compressed particulate phase. Preferably, it is sufficient to inhibit liquid transfer to the extent that after 8 weeks storage immediately after manufacture, at 37°C and at 70% relative humidity at 1 atmosphere pressure, less than 10% by weight, more preferably less than 5% by weight of the material in the compressed solid layer consists of liquid from the semi-solid phase. Thus, after storage at such conditions, the weight of the compressed particulate phase should preferably increase by less than 10%, more preferably by less than 5%.

[0017] The intermediate phase comprises at least 10% by weight of soap preferably at least 15%, more preferably at least 20% and even as high as at least 50% by weight of soap. Typical soap levels in the intermediate phase are from 15% to 50%, preferably from 20% to 40%, more preferably from 25% to 40% by weight. By "soap" is meant one or more inorganic or organic salts of one or more fatty acids, which independently may be saturated or unsaturated and optionally may be branched. Suitable soaps preferably have from 12 to 22 carbon atoms, more preferably from 12 to 18 carbon atoms.

[0018] It is also useful if the intermediate phase is harder than the semi-solid phase and less hard than the compressed particulate phase.

[0019] Preferably the intermediate phase has a weight of from 0.5 to 40 grammes, more preferred from 1 to 20 grammes, most preferred from 2 to 10 grammes. Preferably the other phases each have a weight of 2 to 40 grammes. Preferably the total weight of the cleaning tablet according to the invention is from 10 to 70 grammes.

[0020] The intermediate phase is preferably applied by compaction in a tableting machine and therefore, soap levels substantially higher than 50% by weight (say up to 60% or up to 55% by weight) are less preferred.

[0021] Other ingredients may alternatively or additionally be present in the intermediate phase, although preferably, it 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. Typically such highly soluble materials will have a solubility of at least 100 grammes per litre water of 20° C, more preferred at least 250 grammes.

[0022] The intermediate phase, embodied in the form of a soap containing region of the tablet, may be prepared by any suitable method for example the spraying, applying or brushing of a soap containing formulation, if appropriate followed by hardening e.g. by cooling. In a preferred method, the soap-containing layer is obtainable from the compression of a particulate mixture comprising at least 50% by weight of soap containing particles together with solid particles of any other optional ingredient. The soap containing particles preferably comprise at least 10% by weight, preferably at least 25% 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.

[0023] 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 at least 80 % by weight.

[0024] Further surfactants, for example anionic, nonionic or cationic surfactants may equally be present in the intermediate phase for example at a level of 0.1 to 10 wt% based on the weight of any soap containing part. However, normally, a soap containing phase will be substantially free from non-soap surfactants.

[0025] In addition to the soap surfactants, the intermediate soap rich phase 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, more preferably from 3 to 50 wt%, most preferably 5 to 40 % by weight, based on the weight of the smooth part.

Examples of preferred materials are water-soluble materials such as the sodium and potassium citrates, sodium chloride, sodium sulphate, acetates and carbonates, urea and sugar. The water solubility at 20° C of these materials is preferably at least 10 grammes per 100 ml of water, more preferred more than 15 grammes, most preferably more than 20 grammes.

[0026] If these soluble materials are present, their particle size could be chosen such that the intermediate phase may form as a continuous matrix having dispersed therein particles of the water soluble material.

[0027] 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.

The Semi-Solid Phase

[0028] The semi-solid phase is one which has a consistency which enables some of it to be applied to a fabric by rubbing. Phases with a waxy or soapy or gel-like consistency are especially suitable.

[0029] A preferred semi-solid phase is one which is termed herein, a "smooth phase". Other suitable semi-solid phases are any described in WO-A-00/61717.

[0030] Recently it has been suggested, for example in EP 1,371,719, EP 1,405,900, EP 1,382,668, 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.

[0031] The semi-solid phase preferably contains one or more ingredients which prior to incorporation are liquid at 25°C and 1 atmosphere pressure. This may be selected from one or more aliphatic or aromatic, polar or non-polar organic liquids (particularly organic solvents) such as liquid alkanols, diols and polyols.

[0032] Some particular suitable organic solvents are polyethyleneglycol, dipropyleneglycol, isopropanol or (mono-) propyleneglycol.

[0033] Another preferred class of organic liquids comprises liquid non-soap surfactants such as liquid nonionic surfactants, eg liquid (poly)alkoxylated aliphatic alcohols.

[0034] Preferably, the level of such organic liquids is from 0 to 40 wt%, more preferred 1 to 20, most preferred from 4 to 15 wt% based on the weight of the semi-solid phase.

[0035] The semi-solid phase preferably, however, 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.

[0036] 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.

[0037] Preferably the semi-solid 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.

[0038] The transparency or translucency of a semi-solid phase does not preclude the composition being coloured, e.g. by addition of a dye, provided that it does not detract substantially from clarity.

[0039] In an advantageous embodiment of the invention, the semi-solid 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%.

[0040] Preferably the total weight of surfactants in the semi-solid phase, especially when it is a smooth phase is from 2 to 20 grammes, more preferred from 3 to 10 grammes.

[0041] In a first preferred embodiment of the invention the tablet may be a multi-phase tablet wherein the phases other than the semi-solid phase as described above comprise no or only low levels of non-soap surfactants. Especially the level of non-soap surfactants in the compressed particulate phase is less than 10 wt%(based on the total weight of said phases), more preferred from 0 to 9 wt%, most preferred from 1 to 8 wt%.

The Compressed Particulate Phase

[0042] A compressed particulate phase is a matrix of compacted particles.

[0043] The compressed particulate phase is essentially a tablet portion comprising granular and/or simple powder material compressed into a solid mass. Typically, it will comprise surfactant, preferably also detergency builder and more preferably one or more other ingredients selected from the classes of ingredients used in solid cleaning compositions, especially laundry wash compositions (although other cleaning composition types such as mechanical wavewashing compositions are also possible). Such other optional classes of ingredients may for example be selected from enzymes,

bleaches and bleach systems, fluorescers, antifoams, anti-dye transfer agents, anti-redeposition agents, disintegrants and coloured speckles.

[0044] Although the compressed particulate phase may comprise surfactant materials, this phase preferably comprises ingredients of the tablet 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 this first compressed particulate phase.

[0045] Preferably the first compressed particulate phase is from 5 to 50 grams, for example 10 to 40 grams. Especially preferably the regions are present as layers in the cleaning tablet.

[0046] Preferably the particulate composition has an average 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.

[0047] Tableting to make a compressed particulate phase entails compaction of a particulate composition.

Processing

[0048] Ways of making individual phases have already been described. However, the phases have to be bonded together and/or formed as a unitary mass.

[0049] For making phases or combinations of phases comprising particulate matter, a variety of tableting machinery is known, and can be used. Generally it will function by stamping a quantity of the particulate composition which is confined in a die.

[0050] Manufacture of a tablet with two layers of differing composition may be carried out by placing a predetermined quantity of one composition in a mould, then adding a second composition on top, and next driving a die into the mould to cause compression.

[0051] Alternatively, a predetermined quantity of a first composition may be placed in a mould and compacted by driving a die into the mould, followed by removing the die, adding a second composition and compacting again.

[0052] Tableting machinery able to carry out such operations is known. For example, suitable tablet presses are available from Fette and from Korsch.

[0053] Thus, a two phase tablet comprising the compressed particulate phase and the intermediate phase may be formed such as described above and then the semi-solid phase applied over the intermediate phase, eg by melt adhesion or by bonding with a separate adhesive such as a hot-melt adhesive.

[0054] 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.

[0055] It is known to make tablets using microwave radiation. WO-A-96/06156 mentions that hydrated materials are useful in this special circumstance to cause sintering.

[0056] For the present invention, if any heat is supplied, it is envisaged that this will be supplied conventionally, such as by passing the particulate composition through an oven, rather than by any application of microwave energy.

[0057] The size of a tablet will suitably range from 10 to 160 grams (gm), preferably from 15 to 60 gm, depending on the conditions of intended use, and whether the tablet represents a dose for an average load in a fabric washing 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 is preferably 1040 or 1050 gm/litre, better 1100 gm/litre, up to 1300 or 1350 gm/litre or even more. The tablet density may well lie in a range up to no more than 1250 or even 1200 gm/litre.

[0058] 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. 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.

[0059] Thus the starting particulate composition may suitably have a bulk density of at least 400 g/litre, preferably at least 500 g/litre, and advantageously at least 700 g/litre. Granular detergent compositions of high bulk density prepared by granulation and densification in a high-speed mixer/granulator, as described and claimed in EP-A-340 013, EP-A-352 135, and EP-A-425 277, or by the continuous granulation/densification processes described and claimed in EP-A-367 339 and EP-A-390 251 are inherently suitable for use in the present invention.

Preferred embodiments of the invention will now be described by way of example only. Further modification within the scope of the present invention will be apparent to the person skilled in the art.

[0060] Tablets for use according to the invention are preferably manufactured by a process involving the application of pressure to a particulate mixture. Advantageously the preparation of the soap containing phase may involve the dosing of a particulate mixture comprising ingredients of the intermediate phase, i.e. soap rich particles, preferably in combination

with other materials as described above, followed by the exertion of pressure, preferably above the yield stress of the intermediate phase components. It has been found that the exertion of pressure to a particulate mixture comprising significant levels of soap rich particles leads to a certain flow behaviour of the mixture leading to the formation of an intermediate phase. The semi-solid phase can then be applied by melt-bonding or by use of a suitable adhesive.

[0061] 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).

[0062] Preferably the (co-)compression of the combination of the intermediate and the particulate 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 1- 100 kN/cm², more preferred 2-50 kN/cm², most preferred 2-10 kN/cm².

[0063] The intermediate phase may also be manufactured separately by compression of a suitable composition such as a particulate soap rich material e.g. at the compaction forces as indicated above.

[0064] Alternatively the intermediate phase may be prepared by other methods for example the spraying of a suitable composition such as a soap rich composition, for example onto the (pre) compressed compacted tablet phase on which the intermediate phase layer is formed or adhered. Another suitable method for the preparation of an intermediate phase may involve casting or extrusion of a suitable composition such as a soap containing composition.

[0065] Optionally the semi-solid phase may also be prepared e.g. by extrusion, casting or other shaping methods.

[0066] Separately prepared intermediate phase and semi-solid phase can then be adhered to other part(s) of the tablet for example by gentle pressing or by usage of an adhesive material.

[0067] Similarly a separately prepared solid phase of compressed particulate materials can be combined with one or more pre-prepared intermediate phases e.g. by gentle co-compression, followed by body of the semi-solid phase.

[0068] In general, a fabric washing tablet as a whole will be likely to contain at least 2 wt%, probably at least 5 wt%, up to 40 or 50 wt% surfactant based on the whole tablet, and from 5 to 80 wt% detergency builder, based on the whole tablet.

[0069] Materials which may be used in tablets of this invention will now be discussed in more detail.

Disintegrants

[0070] Preferably, the compressed particulate phase comprises one or more disintegrants.

[0071] Preferred disintegrants are materials which have a solubility in (preferably deionised) water at 20°C of at least 50 grams per 100 grams of water.

[0072] This disintegrant may be present in an amount which is at least 5wt%, 7 wt% or 12 wt% of the tablet. Some of the disintegrant may be present in the base powder used to make the complete tablet formulation used for the compressed particulate phase, whilst the remainder, preferably the majority, is added as a post-dosed ingredient to the base powder before tableting in the compressed particulate phase. It is preferred that at least 75wt% or even 85wt% of the material is not in the base powder, but is added as a post-dosed ingredient.

[0073] A solubility of at least 50 grams per 100 grams of water at 20°C is an exceptionally high solubility: many materials which are classified as water soluble are less soluble than this.

[0074] Some highly water-soluble materials which may be used are listed below, with their solubilities expressed as grams of solid required to form a saturated solution in 100 grams of water at 20°C:

Material	Water Solubility (g/100g)
Sodium citrate dihydrate	72
Potassium carbonate	112
Urea	>100
Sodium acetate (anhydrous)	119
Sodium acetate trihydrate	76
5 Magnesium sulphate 7H ₂ O	71
Potassium acetate	>200

[0075] By contrast the solubilities of some other common materials at 20°C are:

Material	Water Solubility (g/100g)
Sodium chloride	36

(continued)

Material	Water Solubility (g/100g)
Sodium sulphate decahydrate	21.5
Sodium carbonate anhydrous	8.0
Sodium percarbonate anhydrous	12
Sodium perborate anhydrous	3.7
Sodium tripolyphosphate anhydrous	15

[0076] Preferably this highly water-soluble disintegrant is incorporated as particles of the material in a substantially pure form (i.e. the majority of such particles contain over 95% by weight of the material). However, the said particles may contain material of such solubility in a mixture with other material, provided that material of the specified solubility provides at least 50% by weight of these particles.

[0077] The preferred disintegrant of high water-solubility are sodium citrate dihydrate, potassium carbonate, urea, sodium acetate in its anhydrous or trihydrate form, sodium acetate which is partially hydrated - as can be the case when it is spray dried, magnesium sulphate 7H₂O and potassium acetate. Mixtures of these can also be used. The most preferred of the aforementioned materials are sodium citrate dihydrate, sodium acetate in either its anhydrous, trihydrate or partially hydrated form. Mixtures of these most preferred materials can also be used.

[0078] Another suitable class of disintegrants comprises cellulose disintegrants.

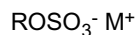
Surfactant Compounds

[0079] 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, zwitterionic, amphoteric, nonionic or a combination of these.

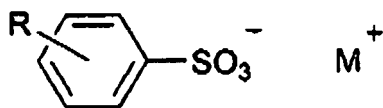
[0080] Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the tablet composition.

[0081] 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.

[0082] 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.

[0083] 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.

[0084] 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.

[0085] Soaps for use in accordance to the invention are 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 C₁₂ to C₂₂ soaps for example from C₁₂ to C₁₈ soaps.

[0086] 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.

[0087] 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.

[0088] 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. 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.

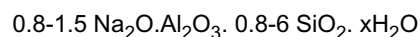
[0089] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition.

[0090] 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.

Detergency Builder

[0091] 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.

[0092] 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:



[0093] 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.

[0094] 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.

[0095] 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.

[0096] 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.

[0097] 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.

[0098] 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

[0099] 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.

[0100] 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, tetraacetylene

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.

[0101] 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

[0102] 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 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.

[0103] The detergent tablets of the invention may also contain (preferably in the second region) a fluorescer (optical brightener), for example, Tinopal (Trade Mark) DMS or Tinopal CBS available from Ciba Ceigy AC, 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.

[0104] An antifoam material is advantageously included (preferably in the second 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.

[0105] 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.

[0106] 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.

[0107] Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the second region) include anti-redeposition 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.

[0108] 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-swelling 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.

Particle Size and Distribution

[0109] When the intermediate phase is embodied as a soap rich region of the tablet, it 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 .

[0110] The intermediate phase is of course a separate phase from the compressed particulate phase which is preferably a matrix of compacted particles.

[0111] Preferably any 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.

[0112] 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. Such tablets have the advantage that, as compared with a

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tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

[0113] 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.

[0114] 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.

[0115] 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.

[0116] The size of a tablet will suitably range from 10 to 160 grams, preferably from 10 to 70g, 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.

[0117] The present invention will now be explained in more detail by way of the following non-limiting examples.

EXAMPLES

[0118] The compressed particulate phase had the following composition:

Composition (%wt)	P1
Na-LAS	4.15
Nonionic 7EO	1.82
Soap	0.33
zeolite A24 (anhydrous)	9.30
Na Acetate.3aq	1.18
Na Carbonate	1.38
SCMC (68%)	0.18
Moisture, salts, NDOM	1.67
Antifoam granule	1.01
Fluorescer granule (15 % active)	2.31
STP HPA	48.1
Nabion/Disilicate co granule.	2.50
TAED (as granule 83%)	4.52
Coated Percarbonate	17.2
Dequest 2047 (43%)	2.70
Enzymes	0.94
Perfume	0.71
TOTAL	100.0

[0119] The following compositions were used for the intermediate phase

Composition wt%	I1	I2
Soap granules (Prisavon 1878 ex Uniqema)	25	50
Granular sodium sulphate	75	50

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[0120] The semi-solid phase had the following composition:

Composition wt%	S1
Na-LAS	32.90
Nonionic 5EO	27.35
Na soap (C16/C18)	2.96
Tween 40	12.05
DiPropyleneGlyc ol	17.77
Dye	0.011
Water	6.96
Total	100

Example 1

[0121] A tablet consisting of a compressed particulate phase, an intermediate phase and a semi-solid phase was produced by first compressing 25 gr of composition P1 in a Fette tableting machine. An intermediate phase was then formed on top of this particulate phase, by compressing 2 gr of composition I1 in the same Fette tableting machine. The semi-solid phase was produced by casting 5 gr of liquid composition S1 at 90°C into a mould, after which the phase was let to solidify. The semi-solid phase was then glued onto the intermediate phase with 0.3 gr of a hot melt water soluble glue, to form the complete tablet.

[0122] With the semi-solid phase a cross was drawn onto the following stains and testcloths:

- WFK10LS (lipstick on cotton)
- WFK10M (motor oil on cotton)
- Ballpoint ink on cotton

[0123] Per stain, roughly 0.3 gr of the semi-solid phase was applied.

The pre-treated stains were subsequently washed with the remaining tablet + one extra tablet with the same composition in a Miele washing machine at 40°C.

In a comparative experiment the test cloths and stains were washed with the same tablets, without using the semi-solid phase to pre-treat the stains.

After the wash the pre-treated areas on the stains were visibly much better cleaned than the untreated areas.

Example 2, comparative Example A

[0124] To demonstrate the necessity of the intermediate phase, tablets were made in the same manner as in Example 1, but now using composition I2 for the intermediate layer (Example 2) and a tablet which did not contain an intermediate layer (comparative Example A).

[0125] The full tablets thus made were flow wrapped and packed in carton boxes and stored for 7 weeks at 37°C and 70% relative humidity.

[0126] After the storage period, the semi-solid phases were removed carefully and re-weighed. The loss in weight (in % of the original gel weight) is a measure of the amount leaked into the compacted particulate phase. The compacted particulate phase was examined visually for changes in colour.

[0127] The table below shows the results:

Example	1	2	A
Weight loss of gel (wt%)	4	6	28
Colour of compacted particulate phase	white	white	Yellow spots

[0128] This result clearly shows the necessity of the intermediate layer to provide stability upon storage, which is an essential feature for these tablets. It also shows that even at fairly low levels of soap (Example 1) surprisingly good barrier properties can be obtained.

Claims

1. Use of a multi-phase laundry cleaning tablet comprising a compressed particulate phase and a semi-solid phase for pretreating a region of a fabric prior to washing by rubbing the semi-solid phase on the region, wherein an intermediate phase is at least partly situated between the semi-solid phase and the compressed particulate phase, and wherein the intermediate phase comprises at least 10% by weight of soap.
2. Use according to any preceding claim, wherein the intermediate phase comprises at least 15%, more preferably at least 20%, preferably at least 50% by weight of soap.
3. Use according to any preceding claim, wherein said intermediate phase is obtainable by compression of a particulate mixture comprising at least 50 wt% of detergent particles, wherein said detergent particles comprise at least 10 wt% of soap surfactant.
4. Use according to any preceding claim, wherein said semi-solid phase comprises up to 40%, preferably from 1% to 20%, more preferably from 4% to 15% by weight of organic liquid.
5. Use according to claim 4, wherein said organic liquid comprises one or more organic solvents selected from liquid alkanols, diols and polyols.
6. Use according to any preceding claim, wherein said semi-solid phase comprises at least 50 wt% (based on the weight of said phase) of non-soap surfactants.
7. Use according to one or more of the preceding claims wherein the semi-solid phase comprises less than 20 wt% of water, preferably 5 to 12 wt%
8. A method of pretreating a region of a fabric prior to washing by rubbing the region with a semi-solid phase of a multi-phase laundry cleaning tablet comprising the semi-solid phase and a compressed particulate phase, wherein an intermediate phase is at least partially situated between the semi-solid phase and the compressed particulate phase, and wherein the intermediate phase comprises at least 10% by weight of soap.
9. A method of washing a fabric, comprising washing the fabric in a wash liquor in which a multi-phase laundry cleaning tablet has been dispersed and/or dissolved, a region of the fabric having first been rubbed with a semi-solid phase of the tablet and wherein the tablet further comprises a compressed particulate phase and wherein an intermediate phase is at least partially situated between the semi-solid phase and the compressed particulate phase, and wherein the intermediate phase comprises at least 10% by weight of soap.

Patentansprüche

1. Verwendung einer mehrphasigen Waschmitteltablette, umfassend eine komprimierte partikuläre Phase und eine halbfeste Phase zum Vorbehandeln eines Bereiches eines Gewebes vor dem Waschen durch Reiben der halbfesten Phase auf den Bereich, wobei sich zwischen der halbfesten Phase und der komprimierten partikulären Phase zumindest teilweise eine Zwischenphase befindet und wobei die Zwischenphase mindestens 10 Gew.-% Seife umfaßt.
2. Verwendung nach einem vorhergehenden Anspruch, wobei die Zwischenphase mindestens 15 Gew.-%, stärker bevorzugt mindestens 20 Gew.-%, am stärksten bevorzugt mindestens 50 Gew.-% Seife umfaßt.
3. Verwendung nach einem vorhergehenden Anspruch, wobei die Zwischenphase durch Kompression eines partikulären Gemisches, umfassend mindestens 50 Gew.-% Waschmittelteilchen, wobei die Waschmittelteilchen mindestens 10 Gew.-% seifenartiges oberflächenaktives Mittel umfassen, erhältlich ist.
4. Verwendung nach einem vorhergehenden Anspruch, wobei die halbfeste Phase bis zu 40, bevorzugt 1 bis 20, stärker bevorzugt 4 bis 15 Gew.-% organische Flüssigkeit umfaßt.
5. Verwendung nach Anspruch 4, wobei die organische Flüssigkeit ein oder mehrere organische Lösungsmittel umfaßt, ausgewählt aus flüssigen Alkanolen, Diolen und Polyolen.

6. Verwendung nach einem vorhergehenden Anspruch, wobei die halbfeste Phase mindestens 50 Gew.-% (bezogen auf das Gewicht der Phase) nichtseifenartige oberflächenaktive Mittel umfaßt.
7. Verwendung nach einem oder mehreren der vorhergehenden Ansprüche, wobei die halbfeste Phase weniger als 20 Gew.-% Wasser, bevorzugt 5 bis 12 Gew.-%, umfaßt.
8. Verfahren zum Vorbehandeln eines Bereiches eines Gewebes vor dem Waschen durch Reiben des Bereiches mit einer halbfesten Phase einer mehrphasigen Waschmitteltabelle, umfassend die halbfeste Phase und eine komprimierte partikuläre Phase, wobei sich zwischen der halbfesten Phase und der komprimierten partikulären Phase zumindest teilweise eine Zwischenphase befindet und wobei die Zwischenphase mindestens 10 Gew.-% Seife umfaßt.
9. Verfahren zum Waschen eines Gewebes, umfassend das Waschen des Gewebes in einer Waschflüssigkeit, in der eine mehrphasige Waschmitteltabelle dispergiert und/oder gelöst worden ist, wobei ein Bereich des Gewebes zuerst mit einer halbfesten Phase der Tablette gerieben worden ist, und wobei die Tablette außerdem eine komprimierte partikuläre Phase umfaßt, und wobei sich zwischen der halbfesten Phase und der komprimierten partikulären Phase zumindest teilweise eine Zwischenphase befindet und wobei die Zwischenphase mindestens 10 Gew.-% Seife umfaßt.

Revendications

1. Utilisation d'une pastille de nettoyage de blanchisserie multi-phase comprenant une phase particulière compressée et une phase semi-solide pour le traitement avant lavage d'une zone d'un textile avant lavage, par frottement de la phase semi-solide sur la zone, dans laquelle une phase intermédiaire est au moins en partie située entre la phase semi-solide et la phase particulière compressée, et dans laquelle la phase intermédiaire comprend au moins 10 % en poids de savon.
2. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle la phase intermédiaire comprend au moins 15 %, de manière davantage préférée au moins 20 %, de manière préférée entre toutes au moins 50 % en poids de savon.
3. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle ladite phase intermédiaire peut être obtenue par compression d'un mélange particulière comprenant au moins 50 % en poids de particules détergentes, dans laquelle lesdites particules détergentes comprennent au moins 10 % en poids de tensioactif savon.
4. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle ladite phase semi-solide comprend jusqu'à 40 %, de préférence de 1 % à 20 %, de manière davantage préférée de 4 % à 15 % en poids de liquide organique.
5. Utilisation selon la revendication 4, dans laquelle le liquide organique comprend un ou plusieurs solvants organiques choisis parmi les alcanols, les diols et les polyols liquides.
6. Utilisation selon l'une quelconque des revendications précédentes, dans laquelle la phase semi-solide comprend au moins 50 % en poids (basée sur le poids de ladite phase) de tensioactifs de type non savon.
7. Utilisation selon l'une ou plusieurs des revendications précédentes, dans laquelle la phase semi-solide comprend moins de 20 % en poids d'eau, de préférence de 5 à 12 % en poids.
8. Procédé de prétraitement d'une zone d'un textile avant lavage, par frottement de la zone avec une phase semi-solide d'une pastille de nettoyage de blanchisserie multi-phase comprenant la phase semi-solide et une phase particulière compressée, dans lequel une phase intermédiaire est au moins en partie située entre la phase semi-solide et la phase particulière compressée, et dans lequel la phase intermédiaire comprend au moins 10 % en poids de savon.
9. Procédé de lavage d'un textile, comprenant le lavage du textile dans une liqueur de lavage dans laquelle une pastille détergente de blanchisserie multi-phase a été de nettoyage et/ou dissoute, une zone du textile ayant été, tout d'abord, frottée avec une phase semi-solide de la pastille et dans lequel la pastille comprend en outre une phase

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particulaire compressée et dans lequel une phase intermédiaire est au moins en partie située entre la phase semi-solide et la phase particulaire compressée, et dans lequel la phase intermédiaire comprend au moins 10 % en poids de savon.

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