(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 1 710 297 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
11.10.2006 Bulletin 2006/41

(21) Application number: **05075810.1**

(22) Date of filing: **08.04.2005**

(51) Int Cl.:
C11D 3/50 *(2006.01)* ***C11D 9/44*** *(2006.01)*
C11D 17/00 *(2006.01)*

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IS IT LI LT LU MC NL PL PT RO SE SI SK TR
Designated Extension States:
AL BA HR LV MK YU

(71) Applicant: **UNILEVER N.V.**
3013 AL Rotterdam (NL)

(72) Inventor: **van den Bergh, Carlo Johannes**
Unilever R&D
3133 AT Vlaardingen (NL)

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

(54) Detergent compositions in tablet form

(57) A method for the production of a cleaning tablet comprising at least two phases, said method comprising the steps of:
(a) preparation of a first phase by compaction of a particulate material, wherein said first phase comprises a perfume material;
(b) preparation of a second phase which is a smooth or non-compressed phase which is substantially free from perfume;
(c) assembly of said first phase and said second phase to produce a cleaning tablet comprising said first phase and said second phase, wherein said first phase comprises a perfume and said second phase is substantially free from perfume;
(d) Incorporating from 1-5 of the cleaning tablets in a pack, wherein said pack is substantially closed and wherein said pack is made from a substantially gas-impermeable material.

EP 1 710 297 A1

Description

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

[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] Several proposals have been made to incorporate perfume in a multi-phase cleaning tablet. WO00/11132 describes the incorporation of perfume in cleaning tablets whereby the perfume is not contained in the same phase with the bleaching agent. WO 99/27069 describes detergent tablets comprising a compressed portion and a 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 perfume.

[0005] It is an object of the present invention to provide a process to prepare a cleaning tablet, comprising at least two phases, wherein said tablet comprises perfume and whereby the perfume does not need to undergo elevated temperatures such that damage to the perfume during processing can be minimised, while on the other hand the perfume is still present in all phases of the tablet such that the user of the tablet can smell the perfume from each phase.

[0006] According to a first preferred embodiment of the present invention there is provided a method for the production of a cleaning tablet comprising at least two phases, said method comprising the steps of:

(a) preparation of a first phase by compaction of a particulate material, wherein said first phase comprises a perfume material;

(b) preparation of a second phase which is a smooth or non-compressed phase which is substantially free from perfume;

(c) assembly of said first phase and said second phase to produce a cleaning tablet comprising said first phase and said second phase, wherein said first phase comprises a perfume and said second phase is substantially free from perfume;

(d) Incorporating from 1-5 of the cleaning tablets in a pack, wherein said pack is substantially closed and wherein said pack is made from a substantially gas-impermeable material.

[0007] The order of steps can be any suitable order provided the resulting product is a pack enclosing 1-5 cleaning tablets each comprising at least 2 phases as described. Preferably steps (a) to (c) take place before step (d). Also suitable order of steps may for example be: (a) and (b) in any order followed by (c) and then (d). Alternatively (b) and (c) or (a) and (c) may take place simultaneously.

[0008] Especially preferable, cleaning tablets made by the process of the invention comprise at least two regions, the first region being a region of a compacted particulate material and the second region being a non-compressed or smooth region.

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

[0010] The regions or phases of a multi-phase tablet are preferably separate layers within a cleaning tablet. However, a discrete region of a tablet could also have other forms for example one or more core(s) or insert(s). In a preferred embodiment the first region is a layer of compacted particulate material and the second region is a smooth layer.

[0011] Preferably the phases in the tablet 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 100 grammes, more preferred from 15 to 60 grammes, most preferred from 15 to 50 grammes.

[0012] Surprisingly it has been found that if cleaning tablets are made as above and subsequently the tablets are stored in a closed, gas-impermeable pack, then the perfume, which initially was incorporated in the compacted phase, migrates during storage via the headspace of the pack into the second smooth phase. This migration leads to a more even distribution of the perfume throughout the tablet thereby creating a pleasant smell,which is observable from all phases of the tablet. Preferably the storage to allow adequate migration of the perfume is at least for a period of 24 hours.

First Phase

[0013] The first compacted phase comprising the perfume.

[0014] In a preferred embodiment of the invention the tablet may be a multi-phase tablet wherein the phases other than the smooth phases as described below comprise no or only low levels of non-soap surfactants. Especially the first phase is a solid phase of compacted materials. Preferably the level of non-soap surfactants in said solid phase is less than 10 %wt (based on the total weight of said phase), more preferred from 0 to 9 %wt, most preferred from 1 to 8 %wt.

[0015] In a first embodiment of the invention the cleaning tablets comprise a first region of compacted particulate material in combination with a second smooth or non-compressed phase.

[0016] Although the first region may comprise some surfactant materials, this region 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 the first region. Preferably the bleach system is predominantly present in the first region. Preferably the enzymes are predominantly present in the first region. 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 first region, more preferred more than 98 %wt, most preferred substantially 100 %wt.

[0017] Advantageously the level of perfume in the first phase is from 0.1 to 20 wt% based on the weight of said phase, more preferred from 0.5 to 15 wt%, most preferably from 1.0 to 10 wt%. The perfume may be added in any form e.g. emulsion, solution, granules, core, encapsulates etc.

[0018] The first phase of the cleaning tablets according to the invention is preferably manufactured by a process involving the application of pressure to a particulate mixture. Advantageously the preparation of the first phase may involve the dosing of a particulate mixture in a mould, followed by the exertion of pressure for example of from 0.1 to 20 kN/cm^2.

Second phase

[0019] In a preferred embodiment of the intention the cleaning tablet comprises in addition to the first phase (as described above) a second phase, which preferably is a non-compressed or smooth phase. This second phase is preferably manufactured by a process involving the heating of a composition which is substantially free from perfume, followed by shaping a cooling said composition. Advantageously the second phase is made by casting or extrusion, whereby a molten composition is shaped followed by cooling. Alternatively the second phase may be prepared by other methods for example the spraying of a composition for example onto the (pre) compressed compacted tablet phase. Another suitable method for the preparation of a soap rich phase may involve casting or extrusion of a composition.

[0020] For the purpose of this invention the term non-compressed or 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.

[0021] The smooth region of the tablet may also contain diluent materials for example polyethyleneglycol, dipropyleneglycol, isopropanol or (mono-)propyleneglycol. Preferable the level of these diluents is from 0 to 40 %wt, more preferred 1 to 20, most preferred from 4 to 15 %wt based on the weight of the smooth phase.

[0022] 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.

[0023] 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.

[0024] 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.

[0025] In an advantageous embodiment of the invention the smooth or non-compressed 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.

[0026] The smooth or non-compressed phase 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.

[0027] The non-soap surfactants in said smooth or non-compressed 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.

[0028] Preferably the smooth or non-compressed phase is prepared by heating a composition to an elevated temperature of from 50 to 125 C, more preferred from 60 to 100 C followed by casting or spraying the composition to form a smooth phase of the desired weight and shape.

Assembly

[0029] The process of the invention involves the preparation of a first phase of compacted particulate material and the preparation of a second smooth or non-compressed phase. Said first and second phases may be prepared separately followed by a additional assembly step. Alternatively the preparation of for example the second phase may inherently also involve the assembly. For example the second phase may be formed by casting or spraying a composition onto the first phase. During this casting or spraying the second phase is prepared while at the same time the first and second phases are assembled into a single multi-phase tablet.

[0030] If the first and second phase are prepared separately, a suitable assembly step may for example involve the gentle pressing of the second phase onto the first phase.

[0031] Optionally adhesive materials may be used for adhering the second phase onto the first phase. Suitable adhesives are for example disclosed in WO 00/52127.

[0032] The above description of the tablet has been given with reference to a tablet constituted by two 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 first region may composed of a limited number (say 1-5) of parts e.g. separate layers in the tablet.

Packaging and storage

[0033] After assembly the cleaning tablets thus obtained is incorporated in a pack which is substantially closed and which is made from a substantially gas-impermeable material. For example the pack may be sealed to ensure no or only limited escape of fragrance from the pack.

[0034] Also preferably the pack may advantageously be made from a substantially non-porous gas or air impermeable material in order to limit the escape of fragrance from the pack. Especially preferably the pack us made from a sealed film or foil material, thereby forming a closed wrapper enclosing 1-5, preferably 1 or 2 cleaning tablets of the invention. A preferred embodiment involves the use of a package in the form of a flow-wrap comprising a tubular plastic film bag having a longitudinal seal 5 and transverse end seals 7.

[0035] Preferably the headspace in pack, i.e. the volume of the pack which is not occupied by the cleaning tablet, is less than 200% based on the volume of the cleaning tablets.

[0036] Advantageously the headspace is from 10 to 150 vol% (based on the volume of the cleaning tablets, more preferred from 20 to 100 vol%. It has been found that a lower volume percentage of the headspace e.g by using a tight packing may lead to insufficient migration of the perfume. Also if the headspace is too large e.g. over 200% based on the volume of the cleaning tablets, then too much perfume may undesirably stay in the headspace rather than migrating into the smooth or non-compressed phase.

[0037] Especially preferably the cleaning tablets may be packed in plastic film e.g a polypropylene or polystyrene film or into a foil material for example a aluminium (coated) foil. Alternatively, though less preferred, the cleaning tablets may be packed in a water-soluble film, for example a PVA film.

[0038] Preferably the incorporation into the pack takes place fairly soon after the preparation and assembly of the first and second phases of the tablet. Normally such incorporation will be within 24 hours, more normally within 1 hour of assembly. After closure of the pack the cleaning tablets of the invention will generally be kept or stored for a certain period for example to transport these to the store and from the store to the home of the user. During this storage, migration of perfume from the first phase into the non-compressed or smooth phase takes place. In order for adequate migration to take place, preferably the packed cleaning tablets are kept or stored for at least 24 hours e.g. during transport from the factory, storage in the shop, transport from the shop to the home etc. Such storage may take place at any suitable temperature e.g ambient temperature.

Use

[0039] 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.

[0040] 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. Alternatively non-phosphate builder materials may be used.

[0041] 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.

[0042] Materials which may be used in tablets of this invention will now be discussed in more detail.

Surfactant Compounds

[0043] 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.

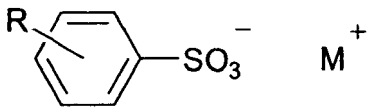
[0044] Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the tablet composition.

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

[0046] Primary alkyl sulphate having the formula

$$ROSO_3^- M^+$$

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



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

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

[0048] 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 occuring fatty acids, preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil. Especially preferably soaps are selected from C_{10} to C_{20} soaps for example from C_{16} to C_{18} or C_{12} soaps.

[0049] 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.

[0050] Specific nonionic surfactant compounds are alkyl (C_{8-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-diamine.

[0051] Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{9-11} and C_{12-15} primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

[0052] 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.

[0053] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition.

[0054] In a machine dishwashing tablet the surfactant may be wholy nonionic, in an amount below 5 wt% of the whole tablet although it is known to include some anionic surfactant and to use up to 10 wt% surfactant in total.

Detergency Builder

[0055] 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.

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

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

[0057] These materials contain some bound water (indicated as "xH2O") and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature. Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in GB 1429143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well known commercially available zeolites A and X, the novel zeolite P described and claimed in EP 384070 (Unilever) and mixtures thereof.

[0058] 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-Na_2SiO_5 morphology form of layered silicate. It can be prepared by methods such as described in DE-A-3,417,649 and DE-A-3,742,043. Other such layered silicates, such as those having the general formula $NaMSi_xO_{2x+1}.yH_2O$ wherein M is sodium or hydrogen, x is a number from 1.9 to 4, preferably 2, and y is a number from 0 to 20, preferably 0 can be used.

[0059] 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.

[0060] 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.

[0061] 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

[0062] 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.

[0063] Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate, advantageously employed together with an activator. Bleach activators, also referred to as bleach precursors, have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example, tetraacetylethylene diamine (TAED), now in widespread commercial use in conjunction with sodium perborate; and perbenzoic acid precursors. The quaternary ammonium and phosphonium bleach activators disclosed in US 4751015 and US 4818426 (Lever Brothers Company) are also of interest. Another type of bleach activator which may be used, but which is not a bleach precursor, is a transition metal catalyst as disclosed in EP-A-458397, EP-A-458398 and EP-A-549272. A bleach system may also include a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

[0064] 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

[0065] The detergent tablets of the invention may also contain (preferably in the second region) one of the detergency enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains. Suitable enzymes include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark.

[0066] 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.

[0067] 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-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.

[0068] 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.

[0069] 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.

[0070] 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.

[0071] Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the second region) include antiredeposition agents such as sodium carboxymethylcellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose, fabric-softening agents; heavy metal sequestrants such as EDTA; and colorants or coloured speckles.

[0072] Further ingredients which can optionally be used in tablets of the invention, preferably in the second region are dispersing aids. Examples of suitable dispersing aids are water-swellable polymers (e.g. SCMC) highly soluble materials (e.g. sodium citrate, potassium carbonate or sodium acetate) or sodium tripolyphospate with preferably at least 40% of the anhydrous phase I form.

Particle Size and Distribution

[0073] The first phase of the cleaning tablet may advantageously be prepared by compacting as described above. Preferably these particles have a mean particle size of from 100 to 1000 μm. Preferably the particulate composition has a mean particle size in the range from 200 to 2000 μm, more preferably from 250 to 1400 μm. Fine particles, smaller than 180 μm or 200 μm may be eliminated by sieving before tableting, if desired, although we have observed that this is not always essential.

[0074] 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.

[0075] 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.

[0076] 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.

[0077] 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.

[0078] 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.

[0079] The present invention will now be explained in more detail by way of the following non-limiting examples.

Example 1

[0080] Preparation of a detergent tablet comprising a first compacted phase, a second smooth phase

Solid compressed phase

[0081] A solid compacted -first- phase was prepared as follows.
A powder was made of the following composition (I) by pre-granulating the granule ingredients, followed by post-dosing the rest of the ingredients.
Composition I:

Ingredient	**Parts by weight**
Linear alkylbenzene sulphonate, sodium salt	8.5
Alcohol ethoxylate nonionic, avg. 7EO	3.7
Zeolite A24	19.0
Sodium acetate, 3aq.	2.4
Sodium carbonate	2.8
Sodium carboxymethyl cellulose	0.4
soap	0.7
Post-dose	
Antifoam adjunct	0.6
Sodium tripolyphosphate	38.1
Sodium di-silicate	2.2
Tetraacetyldiethylenediamine(TAED)	2.9
Sodium percarbonate	10.9
perfume	1.2
Minors (moisture,enzymes)	to 100

[0082] 20 grammes of the particulate composition are inserted into a 45 mm die of a tabletting machine, optionally followed by a flattening step. The whole material is compressed at 60kN into a single tablet.

Smooth second phase preparation:

[0083] The smooth phase was prepared of the following composition:

Ingredient	*Parts by weight*
Na-las	39.1
Nonionic 7EO	33.5
C12 soap	7.3
Monopropyleenglycol	to 100

[0084] The mixture was heated to 80°C and cast into moulds and cooled to 20°C to form firm, 5 grams smooth, parts of 45mm diameter.

[0085] A smooth part is then applied to the top of the tablet e.g. by gentle compression. The resulting tablet is a two-layer tablet whereby initially the perfume is solely located in the compacted layer.

[0086] Two tablets thus obtained are incorporated into a flow-wrap of air impermeable polystyrene film. The size of the flow-wrap is such that the headspace between the tablets and the wrap is about 100% based on the volume of the tablets. The wrapped tablets are stored for 48 hours at ambient temperature.

[0087] Upon removing of the tablets from the wrap it appeared that perfume had migrated from the compacted phase via the headspace into the smooth or non-compressed phase.

Claims

1. A method for the production of a cleaning tablet comprising at least two phases, said method comprising the steps of:

   (a) preparation of a first phase by compaction of a particulate material, wherein said first phase comprises a perfume material;
   (b) preparation of a second phase which is a smooth or non-compressed phase which is substantially free from perfume;
   (c) assembly of said first phase and said second phase to produce a cleaning tablet comprising said first phase and said second phase, wherein said first phase comprises a perfume and said second phase is substantially free from perfume;
   (d) Incorporating from 1-5 of the cleaning tablets in a pack, wherein said pack is substantially closed and wherein said pack is made from a substantially gas-impermeable material.

2. A method according to claim 1 wherein the headspace in the pack is less than 200% based on the volume of the cleaning tablets.

3. A method according to claim 1, wherein the pack is a flow wrap made of a substantially non-porous film or foil.

4. A method according to claim 1 further including the step of storing the packed cleaning tablets for a time sufficiently long to allow migration of the perfume via the headspace from the first phase into the second phase.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 05 07 5810

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
X Y	EP 1 405 902 A (UNILEVER N.V; UNILEVER PLC) 7 April 2004 (2004-04-07) * paragraph [0074]; claims 1-5; examples *	1 1-4	C11D3/50 C11D9/44 C11D17/00
Y	EP 1 340 808 A (HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN) 3 September 2003 (2003-09-03) * paragraphs [0160] - [0167]; claims; examples *	1-4	

TECHNICAL FIELDS SEARCHED (Int.Cl.7)

C11D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	6 October 2005	Loiselet-Taisne, S

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

EPO FORM 1503 03.82 (P04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 05 07 5810

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

06-10-2005

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 1405902 A	07-04-2004	NONE	
EP 1340808 A	03-09-2003	DE 10209157 A1	18-09-2003
		US 2003166492 A1	04-09-2003

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- WO 0142416 A **[0003]**
- WO 0061717 A **[0003]**
- WO 0004129 A **[0003]**
- EP 1371729 A **[0003]**
- EP 1405900 A **[0003]**
- EP 1382368 A **[0003]**
- EP 1375636 A **[0003]**
- EP 1405901 A **[0003]**
- EP 1405902 A **[0003]**
- EP 1418224 A **[0003]**
- WO 03104380 A **[0003]**
- WO 0011132 A **[0004]**
- WO 9927069 A **[0004]**
- WO 0052127 A **[0031]**
- GB 1429143 A **[0057]**
- EP 384070 A **[0057]**
- US 4664839 A **[0058]**
- DE 3417649 A **[0058]**
- DE 3742043 A **[0058]**
- US 4751015 A **[0063]**
- US 4818426 A, Lever Brothers Company **[0063]**
- EP 458397 A **[0063]**
- EP 458398 A **[0063]**
- EP 549272 A **[0063]**
- EP 266863 A, Unilever **[0068]**