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

(11) EP 1 371 719 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
17.12.2003 Bulletin 2003/51

(51) Int Cl.[7]: **C11D 17/00**, C11D 10/04, C11D 3/20

(21) Application number: **03076737.0**

(22) Date of filing: **04.06.2003**

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

(30) Priority: **11.06.2002 EP 02077269**
07.08.2002 EP 02078247

(71) Applicants:
- **UNILEVER N.V.**
  3013 AL Rotterdam (NL)
  Designated Contracting States:
  AT BE BG CH CZ DE DK EE ES FI FR GR HU IT LI LU MC NL PT SE SK TR CY RO SI
- **Unilever Plc**
  London, Greater London EC4P 4BQ (GB)
  Designated Contracting States:
  GB IE

(72) Inventors:
- **Van Doorn, Daniel Albert, Unilever R & D**
  3133 AT Vlaardingen (NL)
- **Nauta, Lammert, c/o Unilever R & D Vlaardingen**
  3133 AT Vlaardingen (NL)
- **Pacha, Fakhruddin Esmail, c/o Unilever R & D**
  3133 AT Vlaardingen (NL)
- **Stuut, Peter Iwan, c/o Unilever R & D Vlaardingen**
  3133 AT Vlaardingen (NL)

(74) Representative: **Joppe, Hermina L. P. et al**
Unilever N.V.,
Patent Department,
P.O. Box 137
3130 AC Vlaardingen (NL)

(54) Detergent tablets

(57) A cleaning tablet and a method for producing a cleaning tablet comprising a smooth or semi-solid phase wherein the smooth or semi-solid phase comprises:

(d) from 10 to 90 wt% of non-soap surfactants; and
(e) from 5 to 90 wt% of soap; and
(f) from 0 to 20 wt% of water

and wherein the production of the smooth or semi-solid phase comprises an extrusion step.

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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 washload.

[0003] 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 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. WO 99/24549 describes a detergent tablet comprising a compressed solid body and a non-compressed gelatinous portion mounted in a mold of said body.

[0005] It is an object of the present invention to provide a method to produce cleaning tablet comprising a smooth or semi-solid phase, wherein said smooth or semi-solid phase comprises surfactants and wherein said cleaning tablet has good dispersing properties of the smooth or semi-solid phase and wherein the smooth or semi-solid phase has a suitable texture (between soft and hard).

[0006] It is a further object of the present invention to provide a method to produce cleaning tablets comprising a smooth or semi-solid transparent or translucent phase, wherein said phase comprises surfactants and wherein said cleaning tablet has good dispersing properties of the smooth or semi-solid phase and wherein the smooth or semi-solid phase has a suitable texture (between soft and hard).

[0007] A further objective of the present invention is to provide a method to produce a smooth or semi-solid tablet or phase thereof wherein the choice of materials and the manufacturing method allows the low cost production of tablets of good performance and of good consistency and texture.

[0008] A further objective of the present invention is to provide a method to produce a smooth or semi-solid tablet or phase thereof wherein the choice of materials and the manufacturing method allows the low cost production of tablets of good performance and of good consistency and texture.

[0009] According to a preferred embodiment of the present invention there is provided a cleaning tablet which has a plurality of discrete regions with differing compositions, characterised in that at least one first region of the tablet is a smooth or semi-solid region and at least one second region of the tablet is a solid region of compacted particulate material.

[0010] In a first aspect the invention relates to a method for producing a cleaning tablet comprising a smooth or semi-solid phase wherein the smooth phase comprises:

(a) from 10 to 90 wt% of non-soap surfactants; and
(b) from 5 to 90 wt% of soap; and
(c) from 0 to 20 wt% of water.

whereas the production of the smooth or semi-solid phase comprises an extrusion step.

[0011] For the purpose of this invention the term "smooth or semi-solid" means that it can be smooth, semi-solid or both smooth and semi-solid.

[0012] Preferably tablets of the invention are of cylindrical shape wherein the two main surfaces (upper side and bottom side) are substantially flat.

[0013] As indicated above, tablets of the invention can be single phase tablets, which are predominantly constituted by the smooth or semi-solid phase as described above. However a preferred embodiment of the invention relates to a multiphase tablet wherein the smooth or semi-solid phase is present and additionally one or more other phases are present. Suitably these additional phases can be smooth, semi-solid or solid. Particularly suitable are solid phases composed of compacted particulate solids.

[0014] The regions of a multi-phase tablet are possibly separate layers within a 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 smooth or semi-solid layer and the second region is a layer of compacted particulate material. In a further advantageous embodiment the first region is a core or insert of smooth or semi-solid material embedded in the second region which is a layer of compacted particulate material.

[0015] If the tablet is a single phase smooth or semi-solid tablet, then preferably the weight of this tablet will be from 5 to 100 g, more preferably from 10 to 40 g, most preferably from 15 to 35 g.

[0016] If the tablet is a multi-phase tablet comprising the smooth or semi-solid phase of the invention then preferably the smooth or semi-solid phase is present as a distinctive region preferably having a weight of from 2 to 20 grammes, more preferred from 3 to 10 grammes. Preferably the other phases together have a weight of 10 to 50 grammes, more preferred 15 to 40 grammes.

[0017] The first region of the tablet preferably is a smooth region. For the purpose of this invention the term smooth 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.

[0018] W099/24549 describes the use of non-compressed gelatinous portions mounted in a mold as a smooth phase. These tablets must be made with specific equipment to ensure the appropriate mold formation. Furthermore the compositions for the smooth phase as disclosed in this document contain very high levels of ingredients with a limited functionality in the wash such as dipropyleneglycolbutylether or glyceroltriacetate.

[0019] WO 00/61717 describes (in the example) the preparation of a compressed particulate tablet on top of which a (non-compressed) smooth layer was made by pouring a mixture of nonionic and PEG followed by hardening. This formulation and its method of preparation is disadvantageous because it requires a very long hardening step in the tablet mould, during which the tablet mould cannot be used for further production, therewith significantly increasing the cost of production.

[0020] Preferably the smooth region of the tablet is also a semi-solid region.

[0021] For the purpose of this invention the term semi-solid refers to compositions which are on the one hand solid enough to retain their shape at ambient temperature but which are not completely solid.

[0022] A suitable test to check if a composition can be considered as semi-solid can be described as follows: A cylindrical tablet with a diameter of 45 mm and a height of 20 mm is compressed radially between the plates of a material testing machine until the tablet fractures. At the starting position, the plates contact the tablet but do not apply force to it. Force is applied to compress the tablet, the vertical speed of the upper plate is 25 mm/minute. The testing machine measures the applied force (F), and also the displacement (x) of the plates towards each other as the tablet is compressed. The distance (y) between the plates before force is applied, which is the diameter of the tablet, is also known. At failure the tablet cracks and the applied force , needed to maintain the displacement, drops. Measurement is discontinued when the applied force needed to maintain the displacement has dropped by 25% from its maximum value.

[0023] A graph of force (F) against displacement (x) is made. The maximum force is the force at failure (F_f). The break energy is the area under the graph of force against displacement, up to the point of break. It is given by the equation:

$$E_b = \int_{0-x_f} F(x)\ dx$$

wherein E_b is the break energy in mJoules, x is the displacement in metres and F is the applied force in Newtons at displacement x and x_f is the displacement at failure.

[0024] Semi-solid compositions are characterised by a ratio of F_f to E_b of less than 1.0, more preferred from 0.1 to 0.9, most preferred from 0.2 to 0.6, while traditional tablets of compacted particulate materials are generally characterised by a ratio of F_f to E_b of more than 1, more generally more than 1.25 or even more than 1.5 up to say 6.

[0025] In an advantageous embodiment of the invention the smooth or semi-solid phase comprises from 20-80 wt% of non-soap surfactants (based on the total weight of said smooth or semi-solid phase), more preferred from 25 to 75 wt%, most preferred 30 to 70 wt%. It has been found that the combination of a separate smooth or semi-solid first region and these high surfactant levels provide very good dispersing and cleaning properties to the tablet.

[0026] Preferably the non-soap surfactants in the smooth or semi-solid region comprise a combination of anionic surfactants and nonionic surfactants in a weight ratio of from 5 : 1 to 1 : 5, more preferred 3 : 1 to 1 : 3, more preferred 2 : 1 to 1: 2. Further surfactants, for example cationic surfactants may equally be present for example at a level of 0.1 to 10 wt% based on the weight of the semi-solid part.

[0027] In addition to the non-soap surfactants the smooth or semi-solid region may comprise soap for example at a level of 2 to 90 wt% based on the weight of the smooth or semi-solid part, more preferred from 3 to 70 wt%, most preferred 5 to 40 wt%.

[0028] It has been found that the soap provides good structuring properties to the smooth or semi-solid phase, especially if this phase comprises relatively high levels of surfactants. This structuring leads on the one hand to a desired firm consistency of the smooth or semi-solid phase but on the other hand retains the smooth or semi-solid nature of the phase. Furthermore the soap is capable of reducing the bleeding of the smooth or semi-solid phase.

[0029] Preferably the smooth or semi-solid 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.

[0030] 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.

[0031] The smooth or semi-solid 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 or semi-solid phase.

[0032] The smooth or semi-solid phase 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 or semi-solid phase, more preferred less than 15 wt%, most preferred from 5 to 12 wt%. Most preferably the smooth or semi-solid 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.

[0033] Preferably the total weight of surfactants in the smooth or semi-solid phase is from 2 to 20 grammes, more preferred from 3 to 10 grammes.

[0034] In a preferred embodiment of the invention the tablet may be a multi-phase tablet wherein the phases other than the smooth or semi-solid phase as described above comprise no or only low levels of surfactants. Preferably the level of surfactants in the the other phases 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%.

[0035] In an advantageous embodiment of the invention the cleaning tablets comprise a first smooth or semi-solid region (as described above) in combination with a second region of the tablet which is a solid region, for example prepared by compression of a particulate composition.

[0036] Although the second region may comprise 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 second region. Preferably the bleach system is predominantly present in the second region. Preferably the enzymes are predominantly present in the second 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 second region, more preferred more than 98 wt%, most preferred substantially 100 wt%.

[0037] 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. For example the first smooth or semi-solid region may be a single discrete part of the tablet but may also be a limited number (say 1-5) discrete smooth or semi-solid parts. Preferably each of these smooth or semi-solid parts are at least 1 gramme, also preferably each of these smooth or semi-solid parts is substantially of the same composition. If reference is made to the composition or weight of the first region it is understood that this concerns the total weight and composition of these smooth or semi-solid parts.

[0038] Similarly the solid second region may be composed of a limited number (say 1-5) of solid parts e.g. separate layers in the tablet. Preferably each of these parts has a weight of at least 10 grammes, also preferably each of the solid parts is substantially of the same composition. If reference is made to the composition or weight of the second region it is understood that this concerns the total weight and composition of these solid parts.

[0039] In addition to the smooth or semi-solid first region and the solid second region the cleaning tablets of the invention may optionally comprise further regions, for example the tablet may be partly or wholly coated.

[0040] Cleaning tablets according to the invention are preferably manufactured by firstly preparing a smooth or semi-solid. Advantageously the preparation of the smooth or semi-solid phase may include the heating of the ingredients followed by cooling. Advantageously the preparation of the smooth or semi-solid phase may involve extrusion.

[0041] Extrusion processes for washing articles are known, for example WO 01/02532 describes the extrusion of washing articles with a pressure of less than 10 bar.

[0042] Preferably extrusion processes for preparing the smooth or semi-solid parts for use in tablets of the invention will involve the forming of an extrudable mass which is then subsequently extruded from an extrusion device and optionally then partioned into parts of the desired size and weight. Optionally the smooth or semi-solid parts may then be hardened.

[0043] The extrudable mass preferably has an initial elevated temperature for example from 60 to 120 C, more preferred from 70 to 90 C. Preferably the extrudable mass is during the production of the smooth or semi-solid mass cooled e.g. to a final temperature of 20 C. If extrusion is used this low temperature may for example be the temperature at the extrusion die for example from 10 to 40, preferably from 15 to 25, most preferred at ambient temperature (20 C).

[0044] In a very preferred embodiment of the invention the conditions for extrusion are carefully controlled. In particular it has been found that smooth or semi-solid parts which on the one hand contain relatively high levels of soap and non-soap surfactants and on the other hand have the right textural properties (i.e not too soft and of semi-solid character) can advantageously be produced by an extrusion process wherein no high-shear conditions are applied, in particular under cooling. Particularly it is preferred that the extrusion takes place under low-shear conditions in the extrusion device. Suitable extrusion devices for this purpose are for example free from extrusion screws. Especially preferred is the feeding of the extrudable mass to a elongated chamber e.g a pipe provided with cooling means but not provided with stirring or shearing devices.A particular preferred low-shear extrusion device is a so-called RAM extruder such as illustrated in figure 1.

[0045] Figure 1 Shows a RAM extruder generally consisting of a tube (1) filled with extrudable mass (9) to be extruded. The extrudable mass is fed into the tube by material feeding means (2). The tube is provided with a movable piston (5) for transporting the extrudable mass through the tube. The piston can be moved by means of a pressing cylinder (3) and retracting cylinder (4). The tube is provided with sleeves for cooling and can be provided, if desired with a die-head.

[0046] The ram extruder generally produces relatively large blocks of smooth or semi-solid material which can be stored and later partioned into smaller parts for tablet assembly.

[0047] After the production of the smooth or semi-solid part the cleaning tablet of the invention may advantageously be made by a process comprising the steps of:

(a) inserting a particulate composition into a tablet mould
(b) inserting one or more smooth or semi-solid parts into said tablet mould
(c) co-compression of the particulate composition and the smooth or semi-solid parts to form a compressed tablet comprising discrete regions, wherein the first region is formed by said smooth or semi-solid parts and the second region is formed by said compressed particulate composition.

[0048] Preferably step (a) takes place before step (b).

[0049] In a preferred embodiment of the invention the particulate composition is pre-compressed at a force of 0.1 to 20 kN/cm^2 between steps (a) and (b) In another preferred embodiment the particulate composition is flattened between steps (a) and (b).

[0050] Preferably the (co-)compression of the combination of the smooth or semi-solid and the solid regions) takes place at a force of from 0.05 to 20 kN/cm^2. Especially if the solid region has been pre-compressed the co-compression in step (c) can advantageously be at a force of 0.1- 10 kN/cm^2, more preferred 0.5 to 5 kN/cm^2. If the solid region has not been pre-compressed, the co-compression preferably takes place at a force of 1- 100 kN/cm^2, more preferred 2-50 kN/cm^2., most preferred 2-10 kN/cm^2.

[0051] One advantage of the method of the present invention is that the co-compression step of (c) leads to good adherence of the first region to the second region and avoids the need of applying an adhesive material between the smooth and semi-solid and solid region. Another advantage of the method of the invention is that it can be carried out in a normal tablet press without the need of adaptation of the shape of the pressing surfaces.

[0052] A tablet of this invention may be intended for use in machine dishwashing. Such a tablet is likely to contain surfactant in a low concentration such as 0.5 to 2 wt% based on the whole tablet, although higher concentrations ranging up to 10 wt% may be used. Such will typically contain salts, such as over 60 wt%, often over 85 wt% of the tablet.

[0053] 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.

[0054] 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% non-soap surfactant based on the whole tablet, and from 5 to 80 wt% detergency builder, based on the whole tablet.

[0055] Materials which may be used in tablets of this invention will now be discussed in more detail.

Surfactant Compounds

[0056] 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 non-soap), cationic, zwitterionic, amphoteric, nonionic or a combination of these.

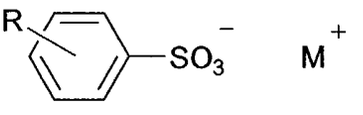
[0057] Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the tablet composition.

[0058] Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

[0059] 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.

[0060] 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 non-soap surfactant in the composition.

[0061] 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.

[0062] 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 C_{10} to C_{20} soaps for example C_{12} soaps.

[0063] 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.

[0064] 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.

[0065] 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.

[0066] 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.

[0067] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition.

[0068] 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

[0069] 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.

[0070] 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:

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

[0071] 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.

[0072] 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.

[0073] 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.

[0074] 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.

[0075] 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.

[0076] 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

[0077] 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.

[0078] 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.

[0079] 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

[0080] The detergent tablets of the invention may also contain (preferably in the second region) one of the detergency enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains. Suitable enzymes include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark. Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in amount of from about 0.1% to about 3.0% by weight of the composition; and these granules or marumes present no problems with respect to compaction to form a tablet.

[0081] 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.

[0082] 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.

[0083] 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.

[0084] 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.

[0085] 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.

[0086] 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

[0087] The second region of a detergent tablet of this invention, is a preferably a matrix of compacted particles.

[0088] 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.

[0089] 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.

[0090] 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.

[0091] 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.

[0092] 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.

[0093] The size of a tablet will suitably range from 10 to 160 grams, preferably from 15 to 60 g, depending on the conditions of intended use, and whether it represents a dose for an average load in a fabric washing or dishwashing machine or a fractional part of such a dose. The tablets may be of any shape. However, for ease of packaging they are preferably blocks of substantially uniform cross-section, such as cylinders or cuboids. The overall density of a tablet preferably lies in a range from 1040 or 1050gm/litre up to 1600gm/litre.

Example 1

[0094] 7 kg of anionic surfactant (Dobanic acid 103 ex Shell) and 6kg of nonionic surfactant (Lutensol AO7 ex BASF) were mixed and neutralised to a pH of 9 using a 50% NaOH solution.

[0095] 10 wt% (based on the weight of neutralised blend) of soap fatty acid (Pristerene 4916 fatty acid ex Uniqema) was added. 5 to 10 wt% (based on the weight of neutralised blend) dipropylene glycol (ex Vopak) was also added to the mixture. The mixture was further neutralised with a 50% NaOH solution to a pH of 11.

[0096] After neutralisation to pH of 11, the mixture was pumped into a sequence of 2 stainless steel tubes by a Maag Sinox P7 pump or a piston pump, type SIBa HK 05016SST4000M000 ex Prominent, Vleuten (NL). Both tubes were double jacketed. The first tube was 2.5m long and had an inner diameter of 73mm. The second tube was 1.5m long and had an inner diameter of 45mm. The tubes were connected by a 10cm long pipe.

[0097] In one experiment the extrusion was performed in the absence of a die-head.In two other experiments a die-head was applied (with inner diameters of resp 24 and 32 mm),which was attatched to the second tube.

[0098] The mixture was pumped into the tubes at a temperature of 85°C at a throughput of 4 kg/hr. The first tube was cooled using a water bath at 40°C. The second tube was cooled using a 50:50 weight mixture of ethylene glycol and water. The coolant temperature was -15°C. The material coming out of the second tube had a temperature of abour 20 C and was collected and divided into bars of around 0.5m.

[0099] After storage the bars were cut into slices of comparable quality of 5 gramme each.

Example II: multi-phase tablets

[0100] A detergent powder was made of the following composition by pregranulating the granule ingredients, followed by post-dosing the rest of the ingredients

Ingredient	*Parts by weight*
Granules	
Na-las	1.1
Nonionic 7EO	0.5
C12 soap	0.1
NaAc.3aq	0.3
Zeolite A24	2.4
Light soda ash	0.4
Moisture/minors	0.4
Post-dose	
EAG (17% silicone)	3.0
Fluorescer (15%)	2.2
STP	62.4
Na-disilicate (80%)	3.8
TAED (83%)	4.3
Percarbonate	16.9
Dequest 2047	1.9
Minors/ enzymes/colour	to 100

[0101] Smooth and semi-solid parts of 5 gramme were prepared as in example 1

[0102] The tablets were made in 2 different ways:

(I) 20 grammes of the powder are inserted into a 45 mm die of a tabletting machine, optionally followed by a flattening step, followed by addition of a single smooth and semi-solid part on top of the powder bed. After addition of the smooth and semi-solid

part onto the powder bed or flattened powder, the whole material is compressed at 30kN into a single tablet, followed by ejection of the tablet. This results in a tablet with a smooth and semi-solid part embedded in the cleaning tablet. The density of the powdered region is 1.5 kg/litre, the density of the semi-solid part is 1.0 kg/litre. The height of the smooth and semi-solid part after compression is 3.4 mm, of the powdered part 11 mm.

(II) Another way of making a tablet with smooth and semi-solid part is to put a single smooth and semi-solid part as above onto 20 grammes of the (optionally pre-compressed at 4 kN) powdered composition in a die of 45 mm diameter followed by a final compression step at 30 kN. During the final compression step, the smooth and semi-solid part flows by the compaction forces to form a 3.4 mm smooth and semi-solid layer adhered on top of the particulate layer. The density of the powdered region is 1.5 kg/litre, the density of the smooth and semi-solid part is 1.0 kg/litre. The height of the powdered part after compression is 11 mm.

Example III

[0103] A detergent powder was made of the following composition by pregranulating the granule ingredients, followed by post-dosing the rest of the ingredients

Ingredient	*Parts by weight*
granules	
Na-las	1.1
Nonionic 7EO	0.5
Soap (C16-C18)	0.1
Zeolite A24	2.4
NaAc3aq	0.3
Light soda ash	0.4
SCMC (68%)	0.1
Moisture/minors	0.4
Post-dose	
EAG (17% silicone)	3.0
Fluorescer (15%)	2.2
STP HPA	28.3
STP LV	34.0
Na-disilicate (80%)	3.8
TAED (83%)	4.3
Percarbonate	16.9
Dequest 2047	1.9
Minors/ enzymes/colour	to 100

[0104] Smooth and semi-solid parts were prepared of the following composition:

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

[0105] The mixture was heated to 80°C and cast into moulds and cooled to 20°C to form firm, 5 grammes smooth and semi-solid parts of 32mm diameter and 6mm high.

[0106] The tablets were made in 2 different ways:

(III) 25 grammes of the powder are inserted into a 45 mm die of a tabletting machine, optionally followed by a flattening step, followed by addition of a single smooth and semi-solid part on top of the powder bed. After addition of the smooth and semi-solid part onto the powder bed or flattened powder, the whole material is compressed at 6 kN/cm^2 into a single tablet, followed by ejection of the tablet. This results in a tablet with a smooth and semi-solid part embedded in the cleaning tablet. The height of the smooth or semi-solid part after compression is 3.4 mm, of the powdered part 11 mm.

(IV) Another way of making a tablet with compressed phase and a smooth and semi-solid part is to put a single smooth and semi-solid part as above onto 25 grammes of the (optionally pre-compressed at 4 kN/cm^2) powdered composition in a die of 45 mm diameter followed by a final compression step at 0.8 kN/cm^2. During the final compression step, the smooth and semi-solid part flows by the compaction forces to form a 3.4 mm smooth semi-solid layer adhered on top of the particulate layer. The height of the powdered part after compression is 11 mm.

[0107] Tablets prepared according the above methods provide good adherence of the smooth or semi-solid part to the rest of the tablet therewith avoiding the need to use adhesive materials. Furthermore the tablets of the invention show fast dispersing of the compacted powder region during the washing process therewith allowing the early release of e.g. builder components into the washing liquor. The compressed semi-solid part shows delayed dispersing therewith providing the surfactants at a later stage during the washing process.

Claims

1. A method for producing a cleaning tablet comprising a smooth or semi-solid phase wherein the smooth phase comprises:

(a) from 10 to 90 wt% of non-soap surfactants; and
(b) from 5 to 90 wt% of soap; and
(c) from 0 to 20 wt% of water

and whereing the smooth or semi-solid phase is manufactured by a process involving extrusion.

2. A method according to claim 1, wherein the extrusion takes place under cooling and/or low-shear conditions.

3. A cleaning tablet comprising a smooth or semi-solid phase comprising

(a)from 10 to 90 wt% of non-soap surfactants; and
(b)from 5 to 90 wt% of soap; and
(c)from 0 to 20 wt% of water

4. A cleaning tablet according to claim 3, wherein the smooth or semi-solid phase has a weight of 2 to 20 grammes.

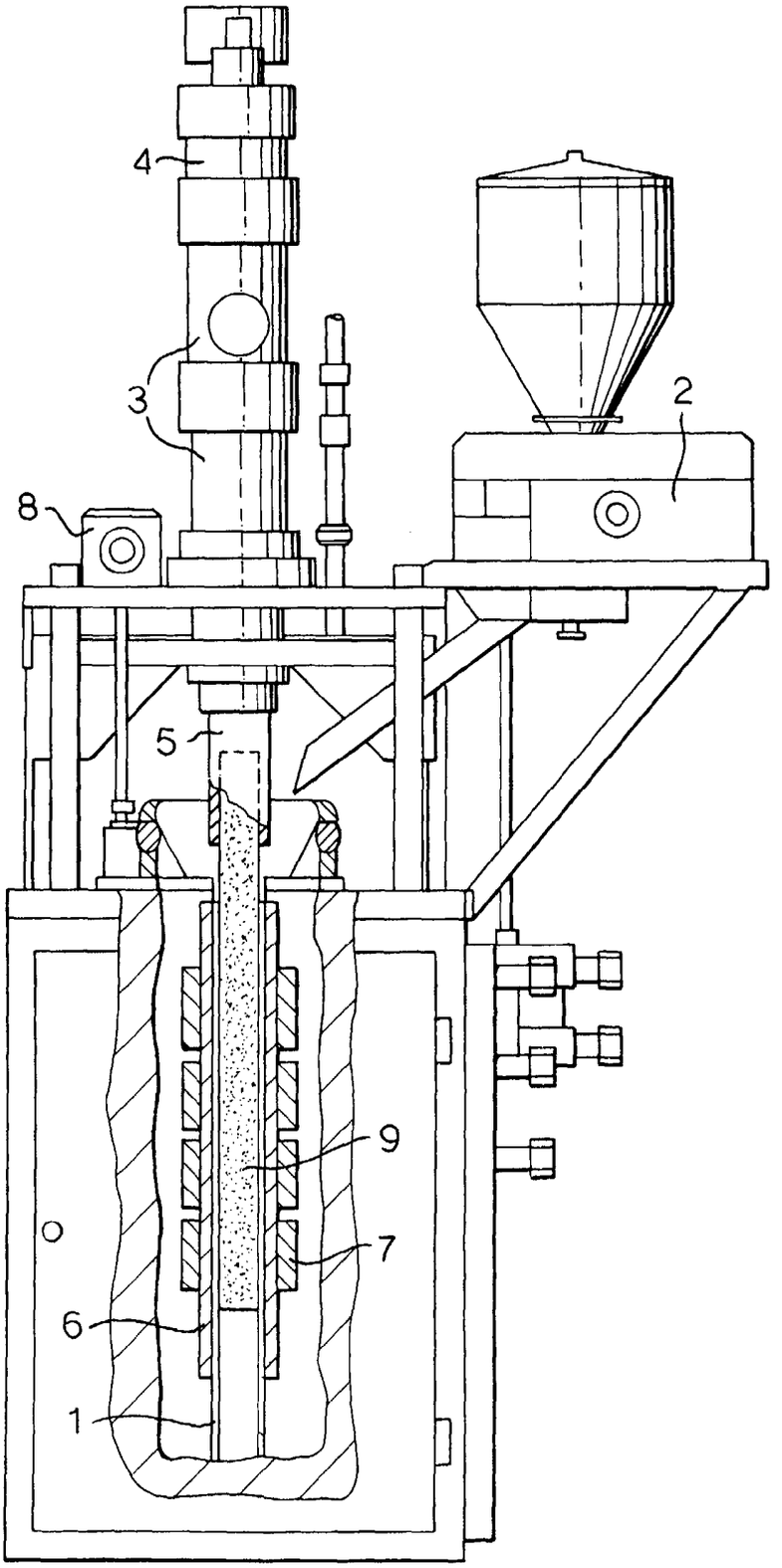
5. A cleaning tablet according to claim 4 wherein the smooth phase is also a semi-solid phase.

6. A cleaning tablet according to claim 3 comprising 5 to 40 wt % soap.

7. A cleaning tablet according to one or more of the preceding claims comprising a smooth or semi-solid region and a solid region.

8. A cleaning tablet according to claim 7 wherein the solid region is a compressed particulate composition.

Fig.1.



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Application Number
EP 03 07 6737

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
MUNICH	18 September 2003	Loloiu, C

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