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(71) Applicants:

UNILEVER N.V.
 3013 AL Rotterdam (NL)

• UNILEVER PLC London EC4P 4BQ (GB)

(72) Inventor: Torn, Lubertus Henricus 3133 AT Vlaardingen (NL)

(74) Representative: Kleiborn, Paul Erik et al Unilever Patent Group Olivier van Noortlaan 120

3133 AT Vlaardingen (NL)

(54) Detergent compositions in tablet form

(57) The invention provides a detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, characterised in that the detergent composition comprises surfactant, especially noni-

onic surfactant, and wherein the majority of the surfactant is in the semi-solid region.

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#### **Description**

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#### Field of the Invention

**[0001]** This invention relates to detergent compositions in the form of shaped bodies, usually referred to as tablets, which are suitable for use in laundry washing, conditioning or softening treatment and/or in automatic dishwashing, which include a solid tablet body and a semi-solid region wherein the majority of surfactant is included.

#### **Background and Prior Art**

**[0002]** Detergent compositions in dishwashing will generally contain a surfactant to be active in the cleaning process. The surfactant generally contributes to soil removal and stabilisation of especially oily soil in the wash water. During the wash process the surfactants may adsorb on the surface of objects to be cleaned which contributes to improved rinsing and drying.

**[0003]** Detergents 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.

**[0004]** Tablets of a cleaning composition are generally made by compressing or compacting a quantity of the composition in particulate form.

[0005] If surfactant is present in the tablet detergent composition it will act mostly in the cleaning phase as soon as the tablet has disintegrated leading to release of the surfactant.

**[0006]** For specific functionality of the surfactant, especially reduction of filming and spotting after and during the rinse phase, it is desired to control the release of surfactant from the detergent composition, especially from a detergent composition in tablet form.

**[0007]** According to WO-A-03/006592 it is desired that the surfactant survives the washing cycle and is predominantly released in the rinse cycle. In WO-03/6592 this is solved by including surfactant in an enclosure that is adapted to be capable of delaying the release of its contents until when or after the temperature in the dishwashing process has reached the cloud point of said surfactant.

**[0008]** The disadvantage of this method of controlled release is that there is no non-ionic surfactant in the cleaning phase and all is released at the start of or during the rinse phase.

[0009] Therefore it is an object of the invention to provide a detergent composition which provides controlled release of surfactant.

#### **Summary of the Invention**

[0010] The present inventors have found that this problem can be solved by a detergent composition in the form of a tablet which includes a semi-solid region, wherein the majority of the surfactant is in the semi-solid region and wherein the level of surfactant in the semi solid region is from 20 to 49 wt%.

**[0011]** The invention provides a detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, wherein the detergent composition comprises surfactant in an amount of from 2 to 8 wt%, and wherein 50 to 100 wt% of the surfactant is present in the semi-solid region and wherein the level of surfactant is from 20 to 49 wt% in the semi-solid region.

#### **Detailed Description of the Invention**

[0012] The detergent composition according to the invention is in the form of a tablet comprising at least a semi-solid region and a solid tablet body.

[0013] It was surprisingly found that the release of surfactant from the semi-solid region is slower than from the solid tablet body.

**[0014]** This extent of release of surfactant into an aqueous medium from the detergent composition may be determined by generally known chromatographic analysis.

**[0015]** To provide the desired cleaning and other functionality at the right moment in the washing- and rinsing cycle, the majority of surfactant is present in the semi-solid region.

**[0016]** For obtaining desired cleaning functionality it is desired that part of the surfactant is released during the washing process and therefore the preferred amount of surfactant in the solid tablet body is from 10 to 40 wt% on total weight of the surfactant.

[0017] In a most preferred embodiment the amount of surfactant present in the semi-solid region is from 60 to 80 wt% on total surfactant.

[0018] The total amount of surfactant in the detergent composition from 2 to 8 wt% on total detergent weight.

**[0019]** Preferably the semi-solid region includes surfactants at a level of 30 to 49 wt% based on the weight of the semi-solid region, more preferred 35 to 45 wt%.

**[0020]** Preferably the level of surfactants in the tablet body is less than 10 wt% (based on the total weight of the tablet body), more preferably from 0.1 to 5 wt%, most preferably from 0.5 to 2 wt% on total weight of the tablet body.

**[0021]** Suitable surfactants are preferably selected from nonionic surfactants and anionic surfactants. Optionally some cationic surfactant is present but this is not preferred for machine dishwash applications.

**[0022]** Highly preferred at least 80%, preferably at least 90%, most preferred all of the surfactant is a nonionic surfactant. In a preferred embodiment all the above weight ranges apply to the nonionic surfactant.

**[0023]** 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.

**[0024]** 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.

**[0025]** 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.

[0026] Preferred non-ionic surfactants for use in machine dishwashing tablets are low to non-foaming nonionic surfactants containing ethylene oxide and/or propylene oxide residues. Examples of suitable low to non-foaming ethoxylated straight-chain alcohols which are preferred nonionic surfactants in machine dishwashing are the Plurafac LF series ex BASF, the Synperonic series ex ICI; the Lutensol® LF series, ex BASF, and the Triton® DF series, ex Rohm & Haas. Also of interest are the end-capped ethoxylated alcohols available as the SLF 18B series from Olin. The dehypon® surfactants ex Cognis are also examples of suitable surfactants.

[0027] Optionally the composition comprises anionic surfactant. Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Preferred examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of  $C_8$ - $C_{15}$ ; olefin sulphonates; alkyl ether sulphates; fatty acids of isethionates; alkane sulphonates; dialkyl sulphosuccinates; fatty acid ester sulphonates, and primary alkyl sulphates having an alkyl chain length of  $C_{8-18}$ .

#### Semi-Solid Region

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[0028] The semi-solid region of the tablet is preferably a separate layer of the tablet. However the region could also have other forms, such as a core or insert.

**[0029]** The semi-solid region is preferably a single discrete part of the tablet but may also be a limited number (for example say 1 to 5) discrete semi-solid parts. Preferably each of these semi-solid parts are at least 1 gram, also preferably each of these semi-solid parts is substantially of the same composition. If reference is made to the composition or weight of the semi-solid region it is understood that this concerns the total weight and composition of these semi-solid parts.

**[0030]** The term "semi-solid" refers to compositions which are one the one hand solid enough to retain their shape at ambient temperature but which are not completely solid.

A suitable test to check if a composition can be considered as semi-solid is described and illustrated in WO03/104380. In this test, a cylindrical tablet of the composition to be tested 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, with the vertical speed of the upper plate being 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. The maximum force is the force at failure (F<sub>f</sub>). 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:

$$E_{b} = \int_{0-x_{f}} F(x) dx$$

in which  $E_b$  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.

**[0031]** Semi-solid compositions are characterised by a ratio of  $F_f$  to  $E_b$  of less than 1.0, more preferably from 0.1 to 0.9, most preferably 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 for example 6.

[0032] The semi-solid region preferably also comprises a soap, more preferred at a level of 2 to 10 wt% based on the weight of the semi-solid region. It was found that the inclusion of soap is preferred to further delay the release of non-ionic surfactant from the semi-solid region. The soap may act as a structurant, forming a network wherein surfactant is present.

[0033] The preferred amount of soap in the semi-solid region is from 4 to 7 wt%.

**[0034]** Any suitable soap may be used, including synthetic soaps, but neutralised fatty acids are highly preferred. The latter were found to lead to desired fine networks that may contribute to the delayed release of surfactant.

**[0035]** Preferably the soap is a (partially) hardened fatty acid soap. Preferred soaps have an iodine value of less than 10, preferably less than 5, more preferred less than 3. The fatty acid chain length is preferably from 16 to 18 carbon atoms. The soaps may be neutralised by any common base such as inorganic base e.g. sodium or potassium hydroxide or organic base.

**[0036]** The preferred soaps are sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

[0037] Suitably the semi-solid region of the tablet comprises diluent materials such as polyethyleneglycol or (mono) or (di-) propyleneglycol. Di-propyleneglycol is a preferred diluent material. Preferably the level of these diluents is from 10 to 70 wt %, more preferably from 15 to 60 wt%, most preferably from 40 to 55 wt% based on the weight of the semi-solid region.

**[0038]** The semi-solid region 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 semi-solid region, more preferably less than 15 wt%, most preferably from 2 to 10 wt%.

#### Solid Tablet Body

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**[0039]** The tablet body according to the invention is a solid which can, for example, be prepared by compression or melting. Preferably the tablet body is a compacted particulate composition.

**[0040]** The solid tablet body is preferably a single discrete part of the tablet but may also be composed of a limited number (for example 1 to 5) of solid parts, such as separate layers in the tablet. Preferably each of these solid parts has a weight of at least 8 grams, also preferably each of these solid parts are substantially of the same composition. If reference is made to the composition or weight of the solid tablet body it is understood that this concerns the total weight and composition of these solid parts.

**[0041]** The solid tablet body preferably comprises ingredients of the tablet other than surfactants. Examples of these ingredients are builders, bleach systems, enzymes etc. Preferably the builders in the tablet are predominantly present in the solid tablet body. Preferably the bleach system is predominantly present in the solid tablet body. Preferably the enzymes are predominantly present in the solid tablet body. For the purpose of this invention the term "predominantly present" refers to a situation in which at least 90 wt% of an ingredient is present in the solid tablet body, more preferably more than 95 wt%, most preferably more than 98 wt%.

### 40 Barrier Layer

[0042] Optionally the detergent composition comprises a barrier layer between the solid tablet body and the semi-solid region.

**[0043]** Preferably, the barrier layer is formed from a hydrated polymeric material interposed between the semi-solid region and the solid tablet body.

**[0044]** Suitable polymeric materials for forming the barrier layer should meet a number of functional requirements, such as good water-solubility, coherent film formation and good adhesion to the semi-solid region and the solid tablet body.

**[0045]** Organic polymeric materials are preferred. Examples of suitable organic polymeric materials include homopolymers and copolymers of vinyl alcohol, homopolymers and copolymers of vinylpyrrolidone, polyethylene glycols, sulfophenol methallyl ether and sodium methallyl sulfonate, and polycarboxylate polymers.

**[0046]** Preferred organic polymeric materials are polycarboxylate polymers.

[0047] Polycarboxylate polymers suitable for forming the barrier layer according to the invention are preferably polymers of unsaturated monocarboxylic acids and/or unsaturated dicarboxylic acids. Suitable monocarboxylic monomers include acrylic, methacrylic, vinylacetic, and crotonic acids; suitable dicarboxylic monomers include maleic, fumaric, itaconic, mesaconic and citraconic acids and their anhydrides. The polymers may also contain units derived from non-carboxylic monomers, preferably in minor amounts.

**[0048]** Examples of suitable polymers are sulphonated terpolymers of acrylic acid, methyl methacrylate and hydrophobically modified polyacrylates.

**[0049]** Polycarboxylate polymers suitable for forming the barrier layer according to the invention generally have a weight average molecular weight from about 1,000 to about 400,000 Daltons.

**[0050]** Especially preferred are homopolymers of acrylic acid and copolymers of acrylic and maleic acids. Commercially available examples of such materials are the acrylic/maleic copolymers available from BASF as the Sokalan (Trade Mark) CP series and the polyacrylates available from BASF as the Sokalan® PA Series. Specific examples are Sokalan® CP5 and preferably Sokalan® PA 25Cl. The polymers are available as powders and granules.

[0051] Mixtures of any of the above described polymeric materials may also be suitable for forming the barrier layer. [0052] The total weight of barrier layer in a tablet according to the invention will preferably range from 0.1 to 15wt%, preferably from 2 to 10wt%, more preferably from 5 to 8wt% (by total weight of barrier layer based on the total weight of the tablet).

#### **Product Forms and Proportions**

[0053] Tablets according to this invention are particularly suitable for laundry or for automatic dishwashing.

[0054] Materials which may be used in tablets of this invention will now be discussed in more detail.

#### Surfactant

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[0055] In an automatic dishwashing composition, surfactant is constitutes from 2 to 8wt%, more preferably from 2 to 6 wt% by total weight of surfactant based on the total weight of the tablet composition, and is likely to consist of nonionic surfactant specified above, either alone or in admixture with anionic surfactant.

**[0056]** The surfactants are further described above.

#### **Detergency Builders**

[0057] Tablets according to the invention or regions thereof may contain a so-called builder which serves to remove or sequester calcium and/or magnesium ions in the water.

**[0058]** The builder is preferably present in the tablet body to enable quick release of builder. Without wishing to be bound by any theory, it is believed that the delayed release of surfactant, especially nonionic-surfactant, from the semi-solid region in a system where the builder is already present may lead to improved cleaning properties of the detergent composition.

[0059] In a most preferred embodiment, from 70 to 100 wt% of the total builder is present in the solid tablet body.

**[0060]** When a water-softening detergency builder is present, the amount of it generally ranges from 5 to 80wt%, more likely from 15 to 80wt% by total weight of detergency builder based on the total weight of the tablet composition.

**[0061]** Non-phosphorus water-soluble water-softening builders may be organic or inorganic. Inorganics that may be present include zeolites, layered silicates, alkali metal (generally sodium) carbonate; while organics 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, nitrilotriacetates and hydroxyethyliminodiacetates.

**[0062]** Tablet compositions preferably include polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers which have some function as water-softening agents and also inhibit unwanted deposition onto fabric from a laundry wash liquor or onto glass from an automatic dishwash liquor.

**[0063]** The category of water-soluble phosphorus-containing inorganic builders includes the alkali-metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates. Specific examples of inorganic phosphate detergency builders include sodium and potassium tripolyphosphates, orthophosphates and hexametaphosphates. Sodium tripolyphosphate is a preferred phosphorus-containing builder for automatic dishwashing tablets. This exists in hydrated, anhydrous or partially hydrated form, and mixtures of these forms may be used to regulate the speed of tablet disintegration and dissolution.

[0064] Preferred water-softening builders are sodium tripolyphosphate, sodium citrate, sodium carbonate and sodium silicate.

## Softening materials

**[0065]** The detergent composition may also comprise softening materials. The term softening material is used herein for purposes of convenience to refer to materials which provide softening and/or conditioning benefits to fabrics in the wash cycle of a home or automatic laundering machine or in a manual wash process.

**[0066]** When the particulate detergent compositions according to the invention comprise softening material, the compositions preferably comprise from 10 to 95% by weight of softening material (active ingredient), based on the total

weight of the composition, more preferably 15 to 75% by weight, most preferably 20 to 50% by weight, e.g. 22 to 45% by weight.

**[0067]** The softening material comprises preferably at least one cationic softening material such as quaternary ammonium fabric softening material. Preferably the quaternary ammonium fabric softening material has two C12-28 alkyl or alkenyl groups connected to the nitrogen head group, preferably via at least one ester link. It is more preferred if the quaternary ammonium material has two ester links present.

**[0068]** Preferably, the average chain length of the alkyl or alkenyl group is at least C14, more preferably at least C16. Most preferably at least half of the chains have a length of C18.

[0069] It is generally preferred that the alkyl or alkenyl chains are predominantly linear.

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[0070] Especially preferred materials are di-alkenyl esters of triethanol ammonium methyl sulphate and N-N-di(tal-lowoyloxy ethyl) N,N-dimethyl ammonium chloride. Commercial examples include Tetranyl AHT-1 (di-hardened oleic ester of triethanol ammonium methyl sulphate 80% active), AT-1(di-oleic ester of triethanol ammonium methyl sulphate 90% active), all ex Kao™. Other unsaturated quaternary ammonium materials include Rewoquat™ WE15 (C10-C20 and C16-C18 unsaturated fatty acid reaction products with triethanolamine dimethyl sulphate quaternised 90 % active), ex Witco™ Corporation.

**[0071]** Other preferred materials include 1,2 bis[tallowoyloxy]-3-trimethylammonium propane chloride and 1,2-bis[oley-loxy]-3-trimethylammonium propane chloride, the method of preparation thereof are, for example, described in US 4137180 (Lever Brothers) of which the contents are incorporated herein. Preferably these materials also comprise small amounts of the corresponding monoester, as described in US 4137180.

[0072] When the detergent composition is to be used as a solid rinse conditioner, the composition may be as described in WO03/083027. Other examples of suitable solid rinse conditioners are described in EP-A-0 234 082, EP-A-0 111 074, EP-A-0 111 074, WO 92/18593, EP-B1-0 568 297, US-A-5 259 964, EP-A-0 107 479 (Unilever), EP-A-0 267 999 (Unilever), JP-A-06 306 769, JP-A-62 057 639 (Lion), JP-A-02 182 972, US-A-4 814 095, GB-A-2 348 435.

[0073] Another class of softening materials are fabric softening clays. In particular those that cooperate with the organic fatty softener materials to provide enhanced softening of laundry. Such clays include the montmorillonite- containing clays which have swelling properties (in water) and which are of smectite structure. The best of the smectite clays for use in the present invention is bentonite and the best of the bentonites are those which have a substantial swelling capability in water, such as the sodium and potassium bentonites. Such swelling bentonites are also known as western or Wyoming bentonites, which are essentially sodium bentonite. Other bentonites, such as calcium bentonite, are normally non-swelling and usually are, in themselves, unacceptable as fabric softening agents.

[0074] However, it has been found that such non-swelling bentonites exhibit even better fabric softening in combination with organic fatty softener materials than do the swelling bentonites, provided that there is present in the softening composition, a source of alkali metal or other solubilising ion, such as sodium (which may come from sodium hydroxide, added to the composition, or from sodium salts, such as builders and fillers, which may be functional components of the composition). Among the preferred bentonites are those of sodium and potassium, which are normally swelling, and calcium and magnesium, which are normally non-swelling. Of these it is preferred to utilise calcium (with a source of sodium being present) and sodium bentonites. The bentonites employed may be produced in the United States of America, such as Wyoming bentonite, but also may be obtained from Europe, including Italy and Spain, as calcium bentonite, which may be converted to sodium bentonite by treatment with sodium carbonate, or may be employed as calcium bentonite. Also, other montmorillonite-containing smectite clays of properties like those of the bentonites described may be substituted in whole or in part for the bentonites described herein and similar fabric softening results will be obtained.

**[0075]** The swellable bentonites and similarly operative clays are of ultimate particle sizes in the micron range, e.g., 0.01 to 20 microns and of actual particle sizes in the range of No's. 100 to 400 sieves, preferably 140 to 325 sieves, U.S. Sieve Series. The bentonite and other such suitable swellable clays may be agglomerated to larger particle sizes too, such as 60 to 120 sieves, but such agglomerates are not preferred unless they include the organic fatty softener materials too (in any particulate products).

[0076] For purposes of providing a treated bentonite in accordance with the invention, the initial bentonite starting material is selected to have relatively low gelling and swelling properties. Specifically, the starting material bentonite is selected to have the following initial properties: (a) a montmorillonite content of at least 85%; and (b) when the bentonite is activated with sodium ions, dried and ground to particles, the ground particles do not swell more than about 2.5 fold over a period of 24 hours when added to deionized water at room temperature. The ground particles of bentonite for purposes of determining swelling herein are particles at least 90% of equal to or less than about 75 microns in diameter. [0077] A detailed description of the process for treating bentonite in accordance with the present invention is disclosed in WO 00/03959 filed in the name of Colin Stewart Minchem, Ltd., the disclosure of which is incorporated herein by reference.

**[0078]** A main component which may be used in combination with the fabric softening clay is an organic fatty softener. The organic softener can be anionic, cationic or nonionic fatty chains ( $C_{10}$  - $C_{22}$  preferably  $C_{12}$  - $C_{18}$ ) Anionic softeners

include fatty acids soaps. Preferred organic softeners are nonionics such as fatty esters, ethoxylated fatty esters, fatty alcohols and polyols polymers. The organic softener is most preferably a higher fatty acid ester of a pentaerythritol compound, which term is used in this specification to describe higher fatty acid esters of pentaerythritol, higher fatty acid esters of pentaerythritol oligomers, higher fatty acid esters of lower alkylene oxide derivatives of pentaerythritol and higher fatty acid esters of lower alkylene oxide derivatives of pentaerythritol oligomers.

**[0079]** Pentaerythritol compound, abbreviated as PEC herein, which description and abbreviation may apply to any or all of pentaerythritol, oligomers, thereof and alkoxylated derivatives thereof, as such, or more preferably and more usually, as the esters, as may be indicated by the context.

[0080] The oligomers of pentaerythritol are preferably those of two to five pentaerythritol moieties, more preferably 2 or 3, with such moieties being joined together through etheric bonds. The lower alkylene oxide derivatives thereof are preferably of ethylene oxide or propylene oxide monomers, dimers or polymers, which terminate in hydroxyls and are joined to the pentaerythritol or oligomer of pentaerythritol through etheric linkages. Preferably there will be one to ten alkylene oxide moieties in each such alkylene oxide chain, more preferably 2 to 6, and there will be one to ten such groups on a PEC, depending on the oligomer. At least one of the PEC OH groups and preferably at least two, e.g., 1 or 2 to 4, are esterified by a higher fatty acid or other higher aliphatic acid, which can be of an odd number of carbon atoms. [0081] The higher fatty acid esters of the pentaerythritol compounds are preferably partial esters. And more preferably there will be at least two free hydroxyls thereon after esterification (on the pentaerythritol, oligomer or alkoxyalkane groups). Frequently, the number of such free hydroxyls is two or about two but sometimes it may by one, as in pentaerythritol tristearate. The higher aliphatic or fatty acids that may be employed as esterifying acids are those of carbon atom contents in the range of 8 to 24, preferably 12 to 22 and more preferably 12 to 18, e.g., lauric, myristic, palmitic, oleic, stearic and behenic acids. Such may be mixtures of such fatty acids, obtained from natural sources, such as tallow or coconut oil, or from such natural air materials that have been hydrogenated. Synthetic acids of odd or even numbers of carbon atoms may also be employed. Of the fatty acids lauric and stearic acids are often preferred, and such preference may depend on the pentaerythritol compound being esterified.

[0082] Examples of suitable detergent compositions containing clay include those described in US-A 6 291 421 and US-A- 6 670 320.

#### **Further Optional Ingredients**

30 [0083] Bleach activators

[0084] Tablets according to the invention may contain bleach activators, also referred to as bleach precursors. Preferred examples include peracetic acid precursors, for example, tetraacetylethylene diamine (TAED), 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 stabiliser (heavy metal sequestrant) may also be present such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

#### Enzymes

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**[0085]** Tablets according to the invention may contain 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 for fabrics 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 Genencor, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark.

**[0086]** Enzymes for dish washing are lipases, amylases and proteases. The enzymes most commonly used in machine dishwashing compositions are amylolytic enzymes. Preferably the composition also contains a proteolytic enzyme. Examples of suitable enzymes are Duramyl® and Theramyl® ex Novozymes.

**[0087]** Detergency enzymes are commonly employed in the form of granules, optionally with a protective coating, in an amount generally ranging from 0.1 to 5.0wt% by total weight of enzyme based on the total weight of the tablet composition.

#### Chelating agent

**[0088]** A chelating agent for metal ions (often referred to as a metal ion sequestrant) may be present in the composition. If present it is preferable that the level of chelating agent is from 0.5 to 3wt% by total weight of chelating agent based on the total weight of the tablet composition. It can function to stabilise bleach against premature decomposition catalysed by transition metals and/or to inhibit calcium carbonate scale formation.

**[0089]** Preferred chelating agents include organic phosphonates, amino carboxylates, polyfunctionally-substituted compounds, and mixtures thereof.

**[0090]** Particularly preferred chelating agents are organic phosphonates such as  $\alpha$ -hydroxy-2 phenyl ethyl diphosphonate, ethylene diphosphonate, hydroxy 1,1-hexylidene, vinylidene 1,1 diphosphonate, 1,2 dihydroxyethane 1,1 diphosphonate and hydroxy-ethylene 1,1 diphosphonate. Most preferred is hydroxy-ethylene 1,1 diphosphonate, 2 phosphono-1,2,4 butanetricarboxylic acid or salts thereof.

**[0091]** Further ingredients which can optionally be employed include anti-tarnishing agents for dishwashing, anti-glass corrosion agents for dishwashing, optical brighteners for laundry, antifoam granules, tablet disintegration aids such as swellable particles or highly water-soluble salts, perfumes, colorants or coloured speckles. Fabric washing tablets may include fabric softeners and/or anti-redeposition agents such as linear sodium carboxymethylcellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose.

Machine dishwashing tablets may include polymers to inhibit filming and polymers to inhibit spotting on glassware.

**[0092]** In a most preferred embodiment the invention relates to a detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, wherein the detergent composition comprises nonionic surfactant, and wherein 50 to 100 wt% of the nonionic surfactant is present in the semi-solid region and wherein the tablet is further characterised in that the total amount of non-ionic surfactant is from 2 to 8 wt% on total weight of the detergent composition, and the semi-solid region comprises from 30 to 49 wt% of nonionic surfactant on the weight of that region and further from 4 to 7 wt% of a soap on the weight of the semi-solid region.

[0093] These compositions preferably comprise a barrier layer as described above.

#### Method of Manufacture

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**[0094]** In another aspect this invention provides a method of making a detergent composition in the form of a tablet, (i.e. shaped body), in which the tablet includes a semi-solid region and a solid tablet body.

[0095] Tablets according to the invention are preferably manufactured by a process comprising the steps of:

- (a) inserting a particulate composition into a tablet mould;
- (b) optionally inserting a barrier in form of inserting an admixture of a polymeric material and a hydrated salt into the tablet mould:
- (c) co-compression of the composition to form a solid tablet body of compacted particulate composition and a barrier layer of hydrated polymeric material, and
- (d) preparing a semi-solid region and applying it to the composition so that the optional barrier layer when present is interposed between the semi-solid region and the solid tablet body.
- [0096] Steps (a) and (b) can be in any order, although preferably the particulate composition is first introduced into the tablet mould, followed by positioning of the admixture of polymeric material and hydrated salt in the mould.
  - **[0097]** Preferably the particulate composition is flattened or pre-compressed before introducing the admixture of polymeric material and hydrated salt (step (b)). Most preferably the particulate composition is pre-compressed at a force of 0.01 to 5 kN/cm<sup>2</sup> before introducing the admixture of polymeric material and hydrated salt.
- [0098] Preferably the co-compression in step (c) takes place at a force of from 0.1 to 20 kN/cm<sup>2</sup>. Especially if the particulate composition has been pre-compressed the co-compression in step (c) can advantageously be at a force of 0.01 to 10 kN/cm<sup>2</sup>, more preferably 2 to 8 kN/cm<sup>2</sup>.
  - **[0099]** The semi-solid region in step (d) may be prepared by mixing of its constituent ingredients. The pressure for preparing the semi-solid region may range from 0 to 5 kN/cm<sup>2</sup>, preferably from 0.01 to 5 kN/cm<sup>2</sup>.
- The application of the semi-solid region in step (d) may take place by spraying or under the application of a co-compression force. Such co-compression may suitably take place at a force of from 0.1 to 20 kN/cm<sup>2</sup>.
  - **[0100]** Tabletting machinery able to carry out the manufacture of tablets of the invention is known, for example suitable tablet presses are available from Carver and from Korsch.
  - **[0101]** The size of a tablet will suitably range from 8 to 160 grams, preferably from 10g to 60g, depending on the conditions of intended use.
  - **[0102]** The tablets may be of any shape such as rectangular, cubic or cylindrical. However, for ease of packaging they are preferably blocks of substantially uniform cross-section, such as cylinders, shapes or cuboids.
  - **[0103]** The overall density of a tablet for fabric washing preferably lies in a range from 1040 or 1050gm/litre preferably at least 1100gm/litre up to 1400gm/litre. The tablet density may well lie in a range up to no more than 1350 or even 1250gm/litre.
  - **[0104]** The overall density of a tablet for machine dishwashing or as a bleaching additive, may range up to 1700gm/ litre and will often lie in a range from 1300 to 1550gm/litre.
  - [0105] The invention will now be illustrated by the following nonlimiting Example, in which all percentages are parts

by weight based on total weight unless otherwise specified.

#### **EXAMPLE**

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<sup>5</sup> **[0106]** A powder composition having the ingredients as shown in Table 1 below was prepared and pre-compressed at a pressure of 0.25 kN/cm<sup>2</sup> on a Carver Laboratory press, to form about 20 gram tablets.

Table 1

Ingredient	wt%
Sodium tripolyphosphate	69.5
Sodium Carbonate	3.0
Non-ionic surfactant (SLF 18B, ex Olin )	1.5
Oxygen bleach	15.0
Sulfonated copolymer	3.5
Bleach activator	2.5
Protease	2.5
Amylase	0.9
Bleach catalyst	1.0
Diphosphonate	1.0
Perfume, Dye	0.1
Total:	100.0

[0107] From these ingredients a tablet was made.

**[0108]** In a further step, the semi-solid composition having the ingredients as shown in Table 2 below was prepared by mixing, heating the mixture to 80°C and spraying into moulds on top of the solid tablet body.

Table 2

Ingredient	Parts by weight
Nonionic surfactant	40
Tallow soap (neutralised by NaOH)	6
Di-propyleneglycol	48
Water	to 100

**[0109]** In the final detergent composition about 24% of the non-ionic surfactant was included in the solid tablet body and about 74% of the non-ionic surfactant was included in the semi-solid region.

**[0110]** When these tablets were used in an automatic diswashing process at about 50 °C, it was found that this resulted in good cleaning and good performance in reduction of spotting and film formation.

#### **Claims**

- 1. The invention provides a detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, wherein the detergent composition comprises surfactant in an amount of from 2 to 8 wt%, and wherein 50 to 100 wt% of the surfactant is present in the semi-solid region and wherein the level of surfactant is from 20 to 49 wt% in the semi-solid region.
- 2. A detergent composition according to claim 1 wherein the surfactant is nonionic surfactant.
- 3. A detergent composition according to any of claims 1-2, wherein the amount of surfactant in the solid tablet body

is from 10 to 40 wt% on total weight of the surfactant.

- 4. A detergent composition according to any of claims 1-3, wherein the semi-solid region comprises a soap.
- 5. A composition according to any of claims 1-4, in which the solid tablet body is a compacted particulate composition.
  - **6.** A composition according to any of claims 1-5 which additionally comprises a barrier layer.
  - 7. A detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, wherein the detergent composition comprises nonionic surfactant, and wherein 50 to 100 wt% of the nonionic surfactant is present in the semi-solid region and wherein the tablet is further **characterised in that** the total amount of non-ionic surfactant is from 2 to 8 wt% on total weight of the detergent composition, and the semi-solid region comprises from 30 to 49 wt% of nonionic surfactant on the weight of that region and further from 4 to 7 wt% of a soap on the weight of the semi-solid region.



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Application Number EP 05 07 7208

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