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(54) **Detergent tablet**

(57) A method of preparing a cleaning tablet which comprises a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a solid region of compacted particulate material and a second region is a smooth region, wherein the method comprises the steps of a) providing a solid region of compacted particulate material thereby forming the first re-

gion; b) fitting tightly an outer sleeve around the periphery of the first region wherein said sleeve extends above the surface area of the first region thereby forming a mould; c) introducing a fluid composition into the mould to form the second region; d) solidifying said second region and optionally removing said sleeve.

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Description

[0001] This invention relates to a process for the preparation of a cleaning composition in the form of tablets. The invention also relates to cleaning compositions in the form of tablets, 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 of a cleaning composition are generally made by compressing or compacting a quantity of the composition in particulate form.

[0004] Recently it has been suggested, for example in 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 phase and a solid phase. However, these tablets require the separate manufacturing of the smooth phase and the solid phase followed by an assembly and packaging step. This renders the manufacturing of these cleaning tablets difficult, time-consuming and costly.

[0005] Therefore it is desirable to have an alternative process or product format which allows the cost-effective production of a detergent tablet comprising a smooth phase and a second phase of compacted particulate material.

[0006] Another problem of the production of tablets comprising a smooth and a compacted phase is the difficulty of achieving adequate adherence of the smooth phase to the compacted phase of the tablet. Often these multi-phase tablets need an adhesive layer to join the more gelatinous smooth layer to the solid compact layer. Alternative methods for preparing multi-phase tablets without adhesive layer have been suggested.

[0007] WO 00/61717 describes the preparation of a compressed particulate tablet on top of which a (non compressed) layer is 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. In addition, the mould needs cleaning after being used.

[0008] WO 99/24549 describes a detergent tablet comprising a compressed solid body and a non-compressed gelatinous portion mounted in a mould of said body. This method requires a pre-prepared mould in the tablet surface, involving extra steps in the process and also special equipment to make such moulds are needed.

[0009] Co-pending non-pre-published European patent application 04077150 describes a method of making a detergent tablet comprising a compressed phase and a smooth phase. The smooth phase is sprayed on the pre-formed compressed phase. In order to have a good surface coverage the spraying involves the formation of

small droplets of the liquid material and the spraying has to be done accurately in order to avoid leakage and spoilage problems. In addition, the spraying sometimes may lead to uneven smooth-phases that make the tablet less attractive to consumers.

[0010] Therefore the present invention aims to provide a process for the production of multi-phase cleaning tablets comprising a compressed phase and a second preferably smooth phase that gives a good adherence of the smooth phase to the compressed phase without the need for an adhesive between said phases, and without the need of a complicated production process and without the need of indents or moulds in the tablet surface and wherein the smooth phase can easily be manufactured and avoiding leakage and spoilage problems.

Description of figure:

[0011] Figure 1 shows a multiphase cleaning tablet of the invention comprising a solid phase (1) and a smooth phase (2) with a sleeve (3). The tablet comprises an upper surface, a bottom surface and a periphery formed by the side(s) connecting said surfaces.

Summary of the invention

[0012] According to a first aspect of the present invention there is provided a method of preparing a cleaning tablet which comprises a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a solid region of compacted particulate material and a second region is a smooth region, characterised in that the method comprises the steps of a) providing a solid region of compacted particulate material thereby forming the first region; b) fitting tightly an outer sleeve around the periphery of the first region wherein said sleeve extends above the surface area of the first region thereby forming a mould; c) introducing a fluid composition into the mould to form said second region; d) solidifying said second region and optionally removing said sleeve.

[0013] In a second aspect, the invention relates to a cleaning tablet comprising a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a solid region of compacted particulate material and a second region is a smooth region, wherein the tablet comprises a sleeve which is tightly fitted around the periphery of the tablet.

Detailed description of the invention

[0014] The sleeve is positioned on the peripheral edges around the solid region of the tablet in such a way that that the sleeve is tightly fitted around the tablet. For the purpose of the invention tightly fitted means that there is essentially no or very little room between the sleeve and the tablet. Preferably the tight fitting ensures that normally the tablet is fixed in the sleeve and no movement of

the tablet region is possible. Suitably the distance between the sleeve and the first region is between 0.1 and 1000 μm , preferably between 0.1 and 100 μm . For the purpose of the invention the solid region has a top- and bottom-surface and side-surfaces which are substantially perpendicular to the top- and bottom-surface. Suitably the side-surfaces are covered by the sleeve. Preferably the sleeve is fitted around the side-surfaces of the solid region whereby the top- and bottom-surface of said region are substantially not covered, such as illustrated in figure 1.

[0015] The sleeve continues above the surface area, preferably the top-surface, of the first region such that the sleeve extends above the surface area thereby forming a mould, such as illustrated in figure 1. Preferably the sleeve extends between 0.1 and 20 mm, more preferably between 1 and 10 mm and most preferably between 1 and 5 mm above the top-surface of the first region.

[0016] After application of the sleeve a fluid is introduced into the mould formed by said sleeve to form the second region. For the purpose of this invention a fluid composition may be a liquid or may have a pasty or gel-like consistency. It may for example be poured, dosed, extruded, or sprayed.

[0017] Preferably the sleeve is made from a film material and is a substantially uniform material. Such film materials may for example be produced by blowing or casting. For the purpose of the invention uniform means that the film has substantially the same composition when comparing one piece of the film with another piece of the film a distance away from the first piece. The film itself however, may consist of more than one layer. Suitable materials are elastomers, water-soluble material, shrink foil and adhesive tape.

[0018] Shrink foil is a material that shrinks upon heating. The advantage of using shrink foil is the very tight wrapping of the sleeve around the tablet thereby preventing leakage of the fluid material between the sleeve and solid region. In addition, the application of the foil does not have to be accurate as the foil will shrink upon application of heat and thereby tightens itself around the tablet.

[0019] Adhesive tape may be convenient as the tape sticks to the solid region and is therefore easy applicable. Double adhesive tape has the further advantage that when the tablet is further packed in a wrapper the tape will also stick to the wrapper. When the tablet is used and the wrapper is removed, simultaneously the tape is also removed and no residue of the tape is left in the wash-load or washing-machine.

[0020] In a preferred embodiment the film is a water-soluble film material. Water-soluble material has the clear advantage that the multi-phase tablet can be directly applied in the washing without leaving a residue after washing. The desired degree of solubilisation and strength can be achieved by matching the type of material and its thickness such that the desired solubilisation time is achieved while still maintaining the desired strength. Also

preferably the film material is deformable under heating conditions.

[0021] Water soluble materials which may be used to form the water soluble films are widely disclosed in the literature and include, for example, polyester amides, polyvinyl alcohol, copolymers of vinyl alcohol and methacrylate, polyethylene oxide, alginates, cellulose ethers such as carboxymethyl cellulose and methylcellulose, starches and starch derivatives, gelatin and any combination of these. Especially preferred is the use of polyvinyl alcohol

[0022] The water-soluble material is preferably mainly composed of poly vinyl alcohol (PVA) or of a co-polymer of poly vinyl alcohol and poly methyl acrylate (PVA-PMA). The term poly vinyl alcohol as used herein also includes partially hydrolysed poly vinyl acetates. The water-soluble film can also contain minor quantities of plasticizers, anti-foams, anti-oxidants, surfactants, perfumes and the like.

[0023] Preferably the film thickness before application to the tablet is from 30 to 150 micrometer, more preferred from 40 to 100 micrometer, most preferred from 60 to 90 micrometer. After application generally the average thickness will be from 10 to 150 micrometers, more preferred from 20 to 100 micrometers, most preferred from 30 to 80 micrometers.

[0024] Preferably the fluid material to be poured into the mould will (just before pouring) have a viscosity of 0.1 Pa.s to 12 Pa.s and a yield stress of 5 to 80 Pa at a shear rate of 21 s^{-1} . These viscosities can for example be determined via the method as described in EP 1,032,642.

[0025] Before the introduction of the smooth phase, preferably the material is pre-heated to an elevated temperature for example from 50 to 150°C , more preferably from 60 to 140°C , most preferably from 70 to 130°C . The fluid, preferably liquid, molten material is then introduced, e.g. poured or dosed into the mould formed by the tightly fitted sleeve.

[0026] Solidification of the fluid material may be accomplished by any suitable method e.g. cooling, gelation or by removal of shear. Preferably the solidification involves the cooling of the fluid phase, preferably to ambient temperature.

[0027] The solid phase of the cleaning tablets according to the invention are preferably manufactured by a process comprising the steps of inserting a particulate composition into a tablet mould and compression of the particulate composition to form a compressed tablet followed by removal of the compressed tablet from the tablet mould.

[0028] In a preferred embodiment of the invention the particulate composition is compressed at a force of 0.1 to 20 kN/cm^2 . After the compression the compressed tablet is preferably removed from the tablet mould.

[0029] Optionally the upper surface of the compressed composition may be treated with one or more materials e.g. barriers before the application of the second region.

[0030] After solidification of the fluid composition, the sleeve may remain on the tablet or may be removed. If the sleeve is to be removed, the fluid material preferably is at least partially solidified, sufficiently to have form retention before the removal of the sleeve.

[0031] Optionally the tablet of the invention may be packed into a suitable packaging material after production. Water soluble film may advantageously be used.

[0032] The use of the process of the present invention has several advantages, pouring the liquid composition on top of the solid region generally leads to a good adherence of the smooth phase to the compressed phase and may generally lead to a reduced or even an absence of the need for an adhesive between the compressed phase and the smooth phase. Another advantage of the process of the present invention is that an expensive metal mould is not needed. For sleeves that remain on the tablet further benefits are that no cleaning steps of the moulding equipment are necessary between two subsequent tablets and that processing times are shortened as there is no need for a waiting time until the fluid phase solidifies before a next tablet is made. Furthermore the presence of a sleeve around the tablet allows the gripping and handling of said tablet without the need of touching the detergent material.

[0033] Also the cleaning tablets produced by a method of the invention generally do not need substantial indentations in its surface (e.g. moulds, indents or cavities) to retain the smooth phase. Generally the upper surface of the compressed phase can be substantially flat, therewith avoiding the need to use complicated equipment to produce said tablet and also avoiding weaknesses in the tablet due to uneven surfaces etc.

[0034] In another preferred embodiment, the present invention relates to a washing tablet comprising a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a solid region of compacted particulate material and a second region is a smooth region wherein the tablet comprises a sleeve which is tightly fitted around the periphery of the tablet. Suitably the distance between the sleeve and the tablet is between 0.1 and 1000 μm . Preferably between 0.1 and 100 μm .

[0035] Suitably the sleeve is made of water-soluble film.

[0036] The sleeve around the tablet has several benefits. The tablets with a sleeve are very appealing to customers; the sleeve gives the tablet a clean look, direct contact between the materials of the tablet and the consumer can be avoided and the tablet is very stable towards breaking and crumbling.

[0037] The regions of the cleaning tablet are possibly separate layers within a tablet. Preferably the second region covers a substantial part of the upper surface of the solid region, e.g. preferably at least 65%, more preferred at least 75%, most preferred more than 90% or even substantially all of the upper surface of the first region is covered by the second region. For the purpose

of the invention the term upper surface refers to one of the main sides of the cleaning tablet which by placing the tablet on a flat surface could be classified as the upper surface. Especially the first region suitably is a layer of compacted particulate material, preferably having a substantially flat upper surface.

[0038] The first region of the tablet is a solid region prepared by compression of a particulate composition.

[0039] Preferably the first region has a weight of from 10 to 50 grams, more preferred from 15 to 40 grams. Preferably the smooth region has a weight of 0.5 to 15 grams, more preferred 1 to 10 grams, most preferred 2 to 6 grams.

[0040] In a preferred embodiment of the invention the solid region of compacted material comprises no or only low levels of surfactants. Preferably the level of surfactants in the first region is less than 10 wt% (based on the total weight of the tablet or even less than 10% wt based on the weight of the first region), more preferred from 0 to 9 wt%, most preferred from 1 to 8 wt%.

[0041] Although the solid 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 solid region. Preferably the bleach system is predominantly present in the solid region. Preferably the enzymes are predominantly present in the solid 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 one region, more preferred more than 98 wt%, most preferred substantially 100 wt%.

[0042] The second region of the tablet is preferably a smooth region. For the purpose of this invention the term smooth phase refers to compositions which are on the one hand solid enough to retain their shape at ambient temperature and on the other hand smooth in appearance. Smooth textures are generally of low or no porosity and have -at normal viewing distance- the appearance of a continuous phase for example as opposed to porous and particulate appearance of a compacted particulate material. Poured smooth regions however may have small irregularities in their upper surface for example caused by uneven solidification or by the inclusion of small air bubbles into the region. Generally however the first region is still smooth in appearance and is clearly distinguishable from the generally uneven appearance of compacted particulate materials.

[0043] Preferably the second region is both a smooth region and a semi-solid region. 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 neither completely solid.

[0044] A suitable test to check if a composition can be considered as semi-solid can be described as follows:

[0045] 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. The displacement at failure (x_f) is also measured.

[0046] A graph of force (F) against displacement (x) can be 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 and 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.

[0047] 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.

[0048] Preferably the second region has an average thickness of from 0.5 to 5 mm, more preferred 1 to 4 mm, for example 1.5 to 3 mm.

[0049] In an advantageous embodiment of the invention the smooth or semi-solid second region comprises from 40-100 wt% of surfactants (based on the total weight of the second region), more preferred from 50-95 wt%, most preferred the second region is predominantly constituted by surfactants e.g. more than 60 wt% for example 70 to 90 wt%. It has been found that the combination of a separate smooth or semi-solid region and these high surfactant levels provide very good dispersing and cleaning properties to the tablet.

[0050] Preferably the total weight of surfactants in the second region is from 0.5 to 10 grams, more preferred from 1 to 5 grams.

[0051] Preferably the surfactants in the second region comprise a combination of anionic surfactants and non-soap non-ionic 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 smooth or semi-solid part. Also advantageously the smooth or semi-solid region may comprise soap for example at a level of 0.1 to 10 wt% based on the weight of the smooth or semi-solid part.

[0052] Also advantageously the second region of the tablet may comprise diluent materials for example poly-ethyleneglycol, (mono-)propyleneglycol or di-propylene glycol. Preferably the level of these diluents is from 0 to 40 wt%, more preferred 2 to 30, most preferred 10-25 wt% based on the weight of the region.

[0053] The second 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 phase, more preferred less than 15 wt%, most preferred from 5 to 12 wt%. Most preferably the second phase is 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.

[0054] 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.

[0055] In addition to the smooth region and the solid compacted region the cleaning tablets of the invention may optionally comprise further regions, for example the tablet may be partly or wholly coated.

[0056] 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 tablet will typically contain salts, such as over 60 wt%, often over 85 wt% of the tablet.

[0057] 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.

[0058] 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% surfactant based on the whole tablet, and from 5 to 80 wt% detergent builder, based on the whole tablet.

[0059] The tablets according to the invention may be placed into a dispensing tray, or preferably directly with the washload into the drum of the washing machine.

[0060] Materials, which may be used in tablets of this invention, will now be discussed in more detail.

Surfactant Compounds

[0061] 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.

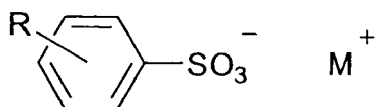
[0062] Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the tablet composition.

[0063] Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

[0064] Primary alkyl sulphate having the formula



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



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

[0065] 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.

[0066] 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.

[0067] It may also be desirable to include one or more soaps of fatty acids. These 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.

[0068] 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.

[0069] Specific nonionic surfactant compounds are alkyl (C₈₋₂₂) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic

C₈₋₂₀ primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-diamine.

[0070] Especially preferred are the primary and secondary alcohol ethoxylates, especially the C₉₋₁₁ and C₁₂₋₁₅ primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

[0071] 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.

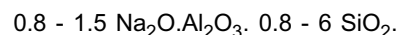
[0072] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition.

[0073] 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

[0074] A composition, which is used in tablets of the invention, will 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.

[0075] 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:



xH₂O

[0076] These materials contain some bound water (indicated as "xH₂O") and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO₂ units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

[0077] 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.

[0078] 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 $\text{NaMSi}_x\text{O}_{2x+1} \cdot y\text{H}_2\text{O}$ 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.

[0079] 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 hexameta-phosphates.

[0080] 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.

[0081] At least one region (preferably the solid region) of a fabric washing tablet preferably includes 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

[0082] Tablets according to the invention may contain a bleach system in at least one region of a tablet, preferably in the smooth 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.

[0083] Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate, advantageously employed together with an activator. Bleach activators, also referred to as bleach precursors, have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example, tetraacetyethylene diamine (TAED), now in widespread commercial use in conjunction with sodium perborate; and perbenzoic acid precursors. The quaternary ammonium and phosphonium bleach activators disclosed in US 4751015 and US 4818426 (Lever Brothers Company) are also of interest. Another type of bleach activator which may be used, but which is not a bleach precursor, is a transition metal catalyst as disclosed in EP-A-458397, EP-A-458398 and EP-A-549272. A bleach system may

also include a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

[0084] 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

[0085] The cleaning tablets of the invention may also contain (preferably in the smooth 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.

[0086] The detergent tablets of the invention may also contain (preferably in the solid 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.

[0087] An antifoam material is advantageously included (preferably in the solid 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.

[0088] It may also be desirable that a cleaning 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.

[0089] A tablet for fabric washing will generally not con-

tain more than 15 wt% silicate. A tablet for machine dish-washing will often contain more than 20 wt% silicate. Preferably the silicate is present in the first region of the tablet.

[0090] Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the solid 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.

[0091] Further ingredients, which can optionally be used in tablets of the invention, preferably in the smooth region are dispersing aids. Examples of suitable dispersing aids are water-swallowable polymers (e.g. SCMC) highly soluble materials (e.g. sodium citrate, potassium carbonate or sodium acetate) or sodium tripolyphosphate with preferably at least 40% of the anhydrous phase I form.

Particle Size and Distribution

[0092] The solid region of a cleaning tablet of this invention is preferably a matrix of compacted particles.

[0093] 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.

[0094] While the starting particulate composition may in principle have any bulk density, the present invention is especially relevant to solid regions made by compacting powders of relatively high bulk density, because of their greater tendency to exhibit disintegration and dispersion problems. Such solid regions 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.

[0095] 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. Tableting machinery able to carry out the manufacture of the solid region of the invention is known, for example suitable tablet presses are available from Fette and from Korch.

[0096] 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.

[0097] 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 dish-washing 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.

[0098] Preferably the diameter of the solid region is substantially the same as the diameter of the smooth region. For the purpose of the invention "substantially the same diameter" means that the diameter of the first phase differs less than 5 mm with the diameter of the second phase, more preferably less than 3mm or even less than 1mm.

Example

[0099] A detergent powder was made of the following composition by pregranulating the granule ingredients, followed by post-dosing the rest of the ingredients

<i>Ingredient</i>	<i>Parts by weight</i>
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

[0100] The tablets were made as follows:

25 grams of the powder are inserted into a 45 mm die of a tableting machine, optionally followed by a flattening step, the material is compressed at 6kN/cm² into a single tablet, followed by ejection of the tablet.

[0101] A ribbon-like piece was cut from a sheet of PVA-film. This 'ribbon' has a height which is slightly larger than the height of the solid phase; in this case the height of the ribbon was around 2 cm. The length of the ribbon was a little longer than the circumference or periphery of

the tablet to provide an overlap for gluing the ribbon together into a sleeve. The length of the ribbon was around 14.5 cm. The ribbon was folded into a circular shape, like a peripheral sleeve and the ends were glued together with water. The diameter of the sleeve was about 2 mm smaller than the diameter of the tablet to obtain a tight fit of the sleeve around the tablet. The sleeve was stretched, and placed around the tablet in a way that the lower edge of the sleeve was at the same height as the bottom of the tablet, and the top of the sleeve extended 3-6 mm above the top surface of the tablet. After releasing, the sleeve shrank to a smaller diameter and was tightly wrapped around the periphery of the solid phase to form a mould.

[0102] Fluid compositions were prepared by mixing the following ingredients:

<i>Ingredient</i>	<i>Parts by weight</i>
Na-las	39.1
Nonionic 7EO	33.5
C12 soap	7.3
Monopropyleenglycol	to 100

[0103] The mixture was heated to 80°C to provide a molten liquid composition. 5 gram of the molten liquid was dosed into the mould formed by the sleeve to form a layer of around 3 mm. The molten liquid was left to solidify for 2 to 3 minutes.

[0104] 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 smooth part shows delayed dispersing therewith providing the surfactants at a later stage during the washing process.

Claims

1. A method of preparing a cleaning tablet which comprises a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a solid region of compacted particulate material and a second region is a smooth region, **characterised in that** the method comprises the steps of

- a) providing a solid region of compacted particulate material thereby forming the first region;
- b) fitting tightly an outer sleeve around the periphery of the first region wherein said sleeve extends above the surface area of the first region thereby forming a mould;
- c) introducing a fluid composition into the mould

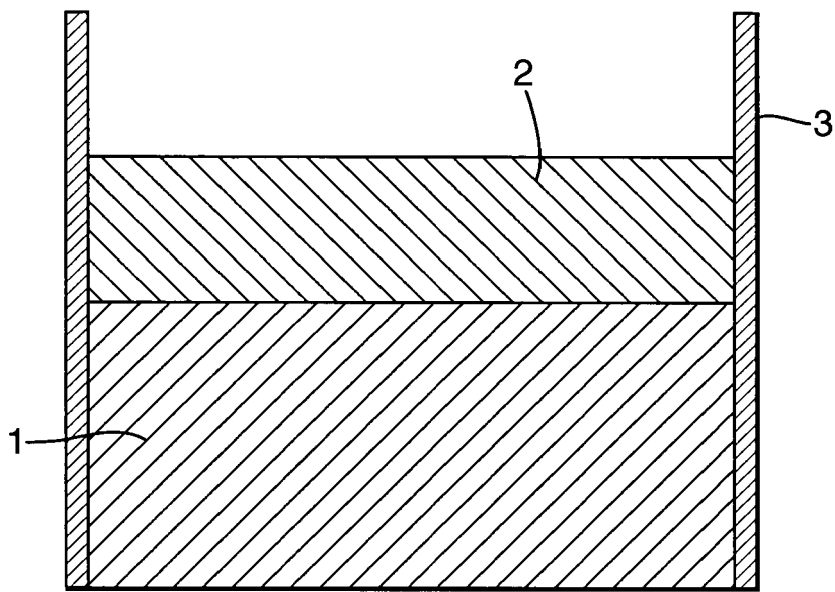
to form the second region;

d) solidifying said second region;

e) optionally removing said sleeve.

2. A method according to claims 1 wherein the first region is substantially free from indents, moulds and cavities.
3. A method according to any of claims 1 to 2, wherein the fluid composition which is introduced into the mould is a molten composition and preferably has a temperature of from 50 to 150 °C, more preferred from 70 to 150 °C.
4. A cleaning tablet comprising a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a solid region of compacted particulate material and a second region is a smooth region, wherein the tablet comprises a sleeve which is tightly fitted around the periphery of the tablet.
5. A cleaning tablet according to claim 4 or a method according to any of the claims 1 to 3 wherein the distance between the sleeve and the tablet is between 0.1 and 1000 µm.
6. A cleaning tablet according to claim 4 or 5 or a method according to any of claims 1 to 3 wherein the sleeve is made of water-soluble film.
7. A cleaning tablet according to any of claims 4 to 6 or a method according to any of claims 1 to 3 wherein the water-soluble film is poly vinyl alcohol (PVA) or of a co-polymer of poly vinyl alcohol and poly methyl acrylate (PVA-PMA).
8. A cleaning tablet according to any of claims 4 to 7 or a method according to any of the claims 1 to 3, wherein the second region comprises 40-100 wt% of surfactants.
9. A cleaning tablet according to any of claims 5 to 8 or a method according to any of the claims 1 to 3 comprising a barrier layer between the first region and the second region.
10. Use of a cleaning tablet according to any of claims 5 to 9 for washing of fabrics or mechanical dishwash.

Fig.1.





European Patent
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Application Number
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