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(54) Process for preparing a cleaning tablet

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

a)placing a sheet of film over a forming die with at least one cavity; b)forming a recess in the film; c)introducing a fluid composition into the recess to form said first region; d)placing a solid composition on top of the first region to form the second region.

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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 EP 1,371,729, EP 1,405,900, EP 1,382,368, EP 1,375,636, EP 1,405,901, EP 1,405,902, EP 1,418,224 and WO 03/104380 to prepare tablets comprising a smooth or semi-solid phase 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 semi-solid 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 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 tables mould cannot be used for further production, therewith significantly increasing the cost of production.

[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-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 preformed 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.

[0011] Surprisingly it was found that a method of placing a sheet of film over a forming die with at least one cavity, forming a recess in the film and introducing a fluid composition into the recess to form a first region, placing a solid composition of compacted particulate material on top of the first region to form a second region, provides for a multi-phase cleaning tablet that does not have the disadvantages of the prior art.

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 has a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a smooth region and a second region is a solid region of compacted particulate material, characterised in that the method comprises the steps of

a)placing a sheet of film over a forming die with at least one cavity;
b)forming a recess in the film;
c)introducing a fluid composition into said recess to form the first region; and
d)placing a solid composition of compacted particulate material on top of the first region to form the second region.

[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 smooth region and a second region is a solid region of compacted particulate material, wherein the tablet has a wrapping of water-soluble film.

[0014] In a third 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 smooth or semi-solid region and a second region is a solid region of compacted particulate material wherein the tablet has a wrapping and wherein the distance of the wrapping to the tablet is between 0.1 and 1000 μm.

Detailed description of the invention

[0015] A suitable device for carrying out the method of the invention comprises a moulding plate. It will preferably comprise a plurality of cavities, which are separated by a distance which is preferably at least 2 mm, more preferred from 10 to 25 mm, most preferred from 11 to 15 mm. Typically, each cavity will have a top surface area of from 0.5 to 50 cm^2, more preferred from 1 to 40 cm^2, most preferred from 1.5 to 35 cm^2. Examples of suitable devices are described in WO 02/16205, WO 00/55045, WO 00/55046, WO 00/55068, WO 00/55069, WO 00/55415 and WO 01/83668.

[0016] Furthermore each cavity, and therefore also the recess in the film, may have various shapes composed of curved or straight lines or combinations thereof for example triangle, hexagonal, round, square, ellips, rectangular. Preferred are rectangular, square or round forms.

[0017] Preferably each cavity or recess has a maximum depth of from 1 to 40 mm, more preferred from 2 to 35 mm, most preferred from 5 to 30 mm.

[0018] Preferably the film material is a substantially uniform material. Such film materials can 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.

[0019] Preferably 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. 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.

[0020] 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, co-polymers 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

[0021] 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.

[0022] Preferably the film thickness to form the recesses (before use, hence in un-stretched form) is from 30 to 150 micrometer, more preferred from 40 to 100 micrometer, most preferred from 60 to 90 micrometer. After stretching 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.

[0023] The recesses are formed when the film is moulded into the cavities of the die. The moulding is suitably performed under heating. In a preferred embodiment, in addition to or alternatively to heating a vacuum is applied to mould the film into the recess. An advantage of applying vacuum is that the film retains firmly into the cavity.

[0024] Preferably the fluid material to be poured into the recesses 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 sheer rate of 21 s-1. These viscosities can for example be determined via the method as described in EP 1,032,642. For the purpose of this invention a fluid composition can be a liquid or have a pasty or gel-like consistency. It may be poured, extruded, or sprayed.

[0025] Before the introduction step c, 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 into the recess.

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

[0027] Preferably the solid composition is placed on top of the fluid composition before the fluid composition solidifies. The solid composition may be placed on top of the fluid material and held there with some pressure. Suitable pressures are 20 to 50 N/cm^2, preferably 40 N/cm^2. When the solid composition is placed on the fluid composition after the fluid is solidified preferably a higher pressure, up to 6000 N/cm^2, preferably between 800 and 4000 N/cm^2, is applied to obtain a good sealing between the solid region and the smooth region.

[0028] 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.

[0029] 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.

[0030] Optionally the upper surface of the compressed composition may be treated with one or more materials e.g. barriers or adhesives before its application on top of the first region.

[0031] After combining the smooth and the compressed regions the film is advantageously sealed for

example by applying a second film on top of the recess containing the detergent composition followed by sealing the first film to the second film. Typical advantageous thickness and materials for the top film may be the same as for the first film.

[0032] Subsequently the sealed packages may be cut to detach from the remaining of the film and from the other packages.

[0033] The use of the process of the present invention has several advantages, pouring the liquid composition and directly placing the solid region on top of the smooth 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 the mould remains clean because of the use of the film material and that this material may directly be used as package material so that there is no need for extra package material and/or cleaning of the mould.

[0034] 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.

[0035] Optionally the wrapped tablet of the invention can be packed into a suitable packaging material after production.

[0036] 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 smooth region and a second region is a solid region of compacted particulate material wherein the tablet has a wrapping of water-soluble film.

[0037] Suitably the wrapping is tightly wrapped around the tablet. In a preferred embodiment the distance of the wrapping to the tablet surface is between 0.1 and 1000 μm.

[0038] In another embodiment the present 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 smooth or semi-solid region and a second region is a solid region of compacted particulate material wherein the tablet has a wrapping and wherein the distance of the wrapping to the tablet is between 0.1 and 1000 μm.

[0039] The distance of the wrapping and the tablet surface may vary within one wrapped cleaning tablet. For instance the distance between the wrapping and the smooth phase may be smaller than the distance between the wrapping and the solid region.

[0040] Suitably the wrapping is made of water-soluble film.

[0041] The wrapping of the tablet has several benefits. The tablets with wrapping are very appealing to customers; the wrapping gives the tablet a clean look, direct contact between the detergent and the consumer is avoided, the tablet is very stable towards breaking and wear and tear and no fragments will break of the tablet. These advantages are especially true for the tight wrapping.

[0042] The regions of the cleaning tablet are possibly separate layers within a tablet. Preferably the first region covers a substantial part of the upper surface of the second 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 second region is covered by the first smooth 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 second region suitably is a layer of compacted particulate material, preferably having a substantially flat upper surface.

[0043] The first 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.

[0044] Preferably the first 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 one the one hand solid enough to retain their shape at ambient temperature but which are neither completely solid.

[0045] A suitable test to check if a composition can be considered as semi-solid can be described as follows:

[0046] 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.

[0047] 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.

[0048] 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.

[0049] Preferably the first region has an average thickness of from 0.5 to 20 mm, more preferred 1 to 5 mm, for example 1.5 to 3 mm.

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

[0051] In an advantageous embodiment of the invention the smooth or semi-solid first 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 first 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 first region and these high surfactant levels provide very good dispersing and cleaning properties to the tablet.

[0052] Preferably the surfactants in the first 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.

[0053] Also advantageously the first region of the tablet may comprise diluent materials for example polyethyleneglycol, (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.

[0054] The first 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 first 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.

[0055] The second region of the tablet is a solid region prepared by compression of a particulate composition.

[0056] Preferably the second region has a weight of from 10 to 50 grams, more preferred from 15 to 40 grams. Preferably the solid region has a weight of 0.5 to 15 grams, more preferred 1 to 10 grams, most preferred 2 to 6 grams.

[0057] 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 second 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 second region), more preferred from 0 to 9 wt%, most preferred from 1 to 8 wt%.

[0058] 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 the second region, more preferred more than 98 wt%, most preferred substantially 100 wt%.

[0059] 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.

[0060] 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.

[0061] 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.

[0062] 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.

[0063] 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% detergency builder, based on the whole tablet.

[0064] The tablets according to the invention may be placed into the dispensing tray, or preferably directly with the washload into the drum of the washing machine.

[0065] Materials, which may be used in tablets of this invention, will now be discussed in more detail.

Surfactant Compounds

[0066] 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.

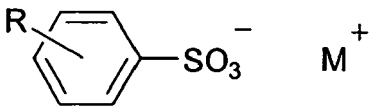
[0067] Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the tablet composition.

[0068] 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.

[0069] 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.

[0070] 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.

[0071] 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.

[0072] 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.

[0073] 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.

[0074] 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.

[0075] 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.

[0076] 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.

[0077] Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition.

[0078] 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

[0079] 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.

[0080] 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:

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

[0081] These materials contain some bound water (indicated as "xH_2O") 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 crys-

talline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

[0082] 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.

[0083] 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.

[0084] 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.

[0085] 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.

[0086] At least one region (preferably the solid 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

[0087] 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.

[0088] 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.

[0089] 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

[0090] 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.

[0091] 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.

[0092] 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.

[0093] 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.

[0094] 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 first region of the tablet.

[0095] Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the solid region) include antiredeposition agents such as sodium carboxymethylcellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose, fabric-softening agents; heavy metal sequestrants such as EDTA; perfumes; and colorants or coloured speckles.

[0096] 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-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

[0097] The solid region of a cleaning tablet of this invention, is a preferably a matrix of compacted particles.

[0098] 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.

[0099] 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.

[0100] 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.

[0101] 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.

[0102] 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.

[0103] 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.

[0104] 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

[0105] 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

Table continued

Post-dose	
TAED (83%)	4.3
Percarbonate	16.9
Dequest 2047	1.9
Minors/ enzymes/colour	to 100

[0106] 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/cm^2 into a single tablet, followed by ejection of the tablet.

[0107] Fluid compositions were prepared by mixing the following ingredients:

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

[0108] The mixture was heated to 80°C to provide a molten liquid composition, which can be poured into the thermo-formed recesses.

[0109] A recess is formed in a sheet using a forming die having a plurality of cavities with dimensions corresponding generally to the dimensions of the tablet to be produced.

[0110] A first sheet of polyvinyl alcohol film (85-micrometer thickness) is drawn over a forming die so that the film is placed over the forming cavities in the die. Each cavity is generally circular shape.

[0111] In order to maximise package strength, the film is delivered to the forming die in a crease free form and with minimum tension. In the forming step, the film is heated to 100 to 120°C, preferably approximately 110°C, for up to 5 seconds preferably approximately 700 micro seconds. A heater is used to heat the film. During this preheating step, a vacuum is pulled through forming die to ensure intimate contact between the film and the preheating plate, this intimate contact ensuring that the film is heated evenly and uniformly (the extent of the vacuum is dependant of the thermoforming conditions and the type of film used). Non-uniform heating results in a formed package having weak spots.

[0112] The thermoformed film is thus molded into the cavities forming recesses which, once formed, are retained in their thermoformed orientation by the application of a vacuum through the walls of the cavities. This vacuum is maintained at least until the packages are filled. Once the recesses are formed and held in position by the vacuum, the heated fluid composition (75°C) is added to each of the recesses. A pre-formed tablet is directly placed on top of the fluid composition and slightly pressed with a force of approx. 320 Newton. At this moment the vacuum can be switched off. Switching of the vacuum has the advantage that the film starts to shrink due to relaxation and thereby presses the fluid composition to the tablet. The tablet is kept in position in the mould for 0.5 to 5 minutes, preferably 0.5 to 1.5 minutes, in which time the gel solidifies. If desired, during this time a slight pressure from the cavities can be applied, pushing the fluid composition to the solid composition. The slight pressure gives excellent bonding of the smooth region to the solid region.

[0113] A second sheet of polyvinyl alcohol film (85-micrometer thickness) is moistened and then superposed on the first sheet covering the filled recesses and adhered thereto. Due to the moistening the second sheet is glued to the first sheet.

[0114] Once sealed, the packages formed are separated from the web of sheet film using cutting means. In this way the packages are formed, filled and sealed while nesting in the forming die. In addition they may be cut while in the forming die as well.

[0115] During the forming, filling and sealing steps of the process, the relative humidity of the atmosphere is controlled at ca. 50%. This is done to maintain the heat sealing characteristics of the film. When handling thinner films, it may be necessary to reduce the relative humidity to ensure that the films have a relatively low degree of plastisication and as such tend to be stiffer resulting in easier handling.

[0116] 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 semi-solid part shows delayed dispersing therewith providing the surfactants at a later stage during the washing process.

Claims

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

    a) placing a sheet of film over a forming die with at least one cavity
    b) forming a recess in the film;
    c) introducing a fluid composition into the recess

to form said first region;
d) placing a solid composition on top of the first region to form the second region.

2. A method according to claim 1 further comprising after step d the steps

e) sealing a second sheet of film across the formed recess to produce at least a closed compartment comprising the cleaning tablet; and
f) optionally cutting in such manner that a package is formed containing the cleaning tablet.

3. A method according to claim 1 or 2 further comprising applying a vacuum and/or heat to the film in step b.

4. A method according to any of claims 1 to 3 wherein step d is carried out before the fluid composition is solidified.

5. A method according to any of claims 1 to 4 wherein the second region is substantially free from indents, moulds and cavities.

6. A method according to claim 5 wherein the upper surface of the second region is substantially flat.

7. A method according to any of claims 1 to 6, wherein the film is a water-soluble film.

8. A method according to claim 7 wherein the water-soluble film is poly vinyl alcohol (PVA) or of a copolymer of poly vinyl alcohol and poly methyl acrylate (PVA-PMA).

9. A method according to any of claims 1 to 8, wherein the fluid composition which is introduced into the recess ins step c)is a molten composition and preferably has a temperature of from 50 to 150 °C, more preferred 70 to 150 °C.

10. A cleaning tablet comprising a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a smooth or semi-solid region and a second region is a solid region of compacted particulate material wherein the tablet has a wrapping of water-soluble film.

11. A cleaning tablet according to claim 10 wherein the distance of the wrapping to the tablet is between 0.1 and 1000 μm.

12. A cleaning tablet comprising a plurality of discrete regions with different compositions, wherein at least a first region of the tablet is a smooth or semi-solid region and a second region is a solid region of compacted particulate material wherein the tablet has a wrapping and wherein the distance of the wrapping to the tablet is between 0.1 and 1000 μm.

13. A cleaning tablet according to claim 12 wherein the wrapping is made of water-soluble film.

14. A cleaning tablet according to any of claims 10 to 13 or a method according to any of the claims 1 to 9 wherein the first region comprises 40-100 wt% of surfactants.

15. A cleaning tablet according to any of claims 10 to 13 or a method according to any of the claims 1 to 9 wherein the first region has a weight of from 0.5 to 15 grams.

16. A cleaning tablet according to any of claims 10 to 13 or a method according to any of the claims 1 to 9 comprising a barrier or adhesive layer between the first region and the second region.

17. A cleaning tablet according to any of claims 10 to 13 or a method according to any of the claims 1 to 9 wherein the first region has an average thickness of from 0.5 to 5 mm.

18. A cleaning tablet according to any of claims 10 to 13 or a method according to any of the claims 1 to 9 wherein the second region has a weight of from 10 to 50 grams.