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(54) **Detergent compositions in tablet form**

(57) A cleaning tablet which has a plurality of discrete regions with differing compositions, characterised in that at least a first region of the tablet comprises an encap-

sulated perfume material embedded in a matrix of a material which is plastically deformable.

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## Description

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

**[0002]** Detergent compositions in tablet form have advantages over powdered products in that they do not require measuring and are thus easier to handle and dispense into the wash-load.

**[0003]** Tablets comprising two or more separate regions have been described. For example, WO 01/42416 describes the production of multi-phase moulded bodies comprising a combination of core moulded bodies and a particulate premix. WO 00/61717 describes a detergent tablet which is characterised in that at least part of its outer surface is semi-solid. WO 00/04129 describes a multi-phase detergent tablet comprising a first phase in the form of a shaped body having at least one mould therein and a second phase in the form of a particulate solid compressed within said mould. Recently, it has been suggested, for example in EP 1,371,729, EP 1,405,900, EP 1,382,368, EP 1,375,636, EP 1,405,901, EP 1,405,902, EP 1,418,224 and WO 03/104380 to prepare tablets comprising a smooth or semi-solid phase, optionally in combination with a solid phase.

**[0004]** A problem with perfume incorporation into cleaning tablets is that often perfume ingredients may suffer from a negative interaction with other components. WO00/11132 describes the incorporation of perfume in cleaning tablets whereby the perfume is not contained in the same phase with the bleaching agent. However, such systems may suffer from bleeding whereby perfume ingredients from one phase of the tablet leak into the other phase. This may still lead to undesirable interaction possibly leading to undesired degradation of ingredients or discoloration of the tablet. WO 99/27069 describes detergent tablets comprising a compressed portion and a non-compressed portion wherein the non-compressed portion comprises a perfume component which could be an encapsulated perfume. However the preparation of a non-compressed phase often involves conditions e.g. elevated temperatures which may lead to damage of the encapsulated perfume.

**[0005]** It is an object of the present invention to provide a method to produce a cleaning tablet comprising at least two phases, wherein said tablet comprises perfume, whereby undesired interaction of perfume ingredients with other components can be minimised. Another object of the invention is to try and minimise leakage of perfume ingredients out of the cleaning tablet thereby maximising the amount of perfume that can be deposited onto the articles to be washed. Surprisingly, it has been found that advantageously perfume can be used in the form of encapsulates, whereby the encapsulates are incorporated into a separate phase and whereby preferably said phase is produced by a process which avoid high temperatures and high pressures.

**[0006]** According to a first preferred embodiment of the present invention, there is provided a detergent tablet which has a plurality of discrete regions with differing compositions, characterised in that at least a first region of the tablet comprises an encapsulated perfume material embedded in a matrix of a material which is plastically deformable under pressure.

**[0007]** In a preferred aspect, the invention also relates to a method for producing a detergent tablet wherein at least a first region of the tablet comprises an encapsulated perfume material embedded in a matrix of a material which is plastically de-formable under pressure, wherein the production of said region comprises the compression of a particulate composition comprising at least 10 %wt of a substance which is plastically de-formable under pressure.

**[0008]** Especially preferably, tablets of the invention comprise at least three regions, the first region comprising the perfume encapsulate embedded in a matrix of a material which is plastically de-formable under pressure, the second region being a smooth region and the third region being a region of a compacted particulate material.

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

**[0010]** As indicated above, tablets of the invention comprises a first phase comprising an encapsulated perfume embedded in a matrix of a material which is plastically de-formable under pressure. One preferred arrangement is to utilize the invention for the middle layer of a tablet with three layers. A further advantage of this is that the plastically de-formable material acts as a binder, and promotes adhesion of the two outer layers to the middle layer. Preferably additionally the tablet comprises a smooth phase and a compressed phase of compacted particulate materials. Most advantageous is the location of the first phase as a barrier between the compressed phase and the smooth phase.

**[0011]** The regions or phases of a multi-phase tablet are preferably separate layers within a detergent tablet. However, a discrete region of a tablet could also have other forms for example one or more core(s) or insert(s). In a preferred embodiment the first region is a layer of a material which is plastically de-formable under pressure and the second region is a smooth layer and a third layer is a layer of compacted particulate material. Preferably the first layer is located substantially as a barrier between the second and third layer.

**[0012]** If the tablet is a multi-phase tablet comprising the encapsulated perfume embedded in a plastically de-formable material phase of the invention, then preferably this first phase is present as a distinctive region preferably having a weight of from 0.5 to 15 grammes, more preferred from 1 to 10 grammes, most preferred from 1.5 to 8 grammes. Preferably the other phases in the tablet each have a weight of 2 to 40 grammes. Preferably the total weight of the

detergent tablet according to the invention is from 10 to 100 grammes, more preferred from 15 to 60 grammes, most preferred from 15 to 50 grammes.

#### First phase

**[0013]** The first phase comprising the encapsulated perfume embedded in the plastically de-formable material may be prepared by any suitable method e.g. mixing, casting, spraying etc, however in a preferred method said first phase is obtained from the compression of particles comprising at least 10wt% of a de-formable substance. Such particles preferably comprise at least 10 %wt, more preferably at least 50%wt (based on the particles) of the plastically de-formable substances.

**[0014]** For the purpose of the invention the term "material which is plastically deformable under pressure" refers to any material which on the one hand can exist in a solid form or particulate form under ambient pressure and at ambient temperatures of say 15 to 25°C and preferably somewhat above, but which on the other hand under moderate (gauge) pressure of say from 0.1 to 10, especially from 0.2 to 2 kN/cm<sup>2</sup> can merge or flow together, e.g. when subjected to the compaction pressure.

**[0015]** Preferably, the plastically de-formable material has a softening or melting temperature above ambient, conveniently above 35°C, better at least 40°C. Preferably, the melting temperature does not exceed 80°C, or even 70°C. Also preferably, the plastically de-formable material is water-soluble. A solubility of at least 10g per 100g de-ionized water at 20°C is desirable. A higher solubility, such as at least 20g per 100 g de-ionized water at 20°C, is preferred.

**[0016]** Preferred plastically de-formable substances for inclusion in the plastically deformable material are for example organic polymers containing polar groups, especially polyethylene glycol (PEG). Polyethylene glycols of molecular weight from 1000 to 10,000 and above have been found suitable, especially those of molecular weight in a range from 1000 to 5000. Other plastically de-formable organic polymers with a high proportion of hydrophilic groups could be employed. Possibilities are polyacrylates and polyvinyl pyrrolidone. The plastically de-formable substance may have surfactant properties for example nonionic surfactants containing an average of 20 or more (preferably 30 or more) ethylene oxide residues, anionic surfactants for example particles of linear alkyl benzyl sulphonate and soap particles.

**[0017]** Preferably the level of plastically de-formable substance in the first phase is from 10 to 90 wt%, more preferred from 20 to 85 wt%, most preferred from 30 to 70 wt% based on the weight of the first phase.

**[0018]** Advantageously the first phase may further comprise water-soluble, solid material in addition to the plastically deformable substance used to form the matrix embedding the encapsulated perfume, for example solid non-deformable materials such as soluble mineral salts, bleaches etc. Preferably such solid materials are hard materials which generally are not plastically deformable. The level of these solid materials may suitable range from 10 to 90 wt% based on the total weight of the first phase of the tablet, for example from 15 to 75wt%. Preferred solid materials include water soluble materials such as electrolyte materials, meltable organic materials and sugars. Examples of preferred materials are water-soluble materials such as for example sodium and potassium citrates, sodium and potassium chloride, sodium or potassium acetates, alkali metal sulphates or carbonates, urea and sugar. The water solubility at 20 C of these materials is preferably at least 10 grammes per 100 ml of water, more preferred more than-15 grammes, most preferably more than 20 grammes.

**[0019]** If these solid soluble materials are present, their particle size is preferably chosen such that the phase is a continuous matrix of the plastically de-formable material having dispersed therein particles of the water soluble material.

**[0020]** It has been found that these solid materials provide good dissolution properties to the first phase. Furthermore these materials do not negatively affect the desired firm consistency of said phase.

#### Encapsulated perfume

**[0021]** The first phase of the tablet also comprises an encapsulated perfume material. Perfume encapsulates preferably of the are core in shell type such as for example described in GB 0751600, EP 385,534, US 3,341,466 and which are commercially available. Preferred perfume encapsulates are melamine-formaldehyde-urea capsules for example as described in US 6,224,795, US 3,516,941 and US 5,154,842. Encapsulated perfumes may be added as such to the particles forming the first phase, but may can also advantageously be used e.g as slurries which are applied to particulate materials to be used in the first phase, for example the perfume encapsulates may be applied to cores which comprises for example one or more detergent materials such as surfactants or builders, whereby advantageously said cores comprise from 1 to 10 wt% of perfume encapsulates. A suitable process for producing said granules or particulates is for example disclosed in our non prepublished application EP03079123.

**[0022]** Advantageously, the level of perfume in the first phase is from 0.1 to 20 wt% based on the weight of said phase, more preferred from 1 to 15 wt%. If the perfume is added as part of cores to which the encapsulated perfumes are applied e.g. in amounts up to 10 wt% based on said cores, then preferably the level of such coated cores is preferably from 5 to 50 wt% based on the weight of the first phase, more preferred from 10 to 40 wt%.

Second phase

**[0023]** In a preferred embodiment of the invention, the detergent tablet comprises in addition to the first phase (as described above) a second phase, which is a smooth phase.

**[0024]** 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.

**[0025]** The smooth region of the tablet may also contain diluent materials for example polyethyleneglycol, dipropyl-ene glycol, isopropanol or (mono-)propyleneglycol. Preferably the level of these diluents is from 0 to 40 %wt, more preferred 1 to 20, most preferred from 4 to 15 %wt based on the weight of the smooth phase.

**[0026]** The smooth phase comprises no or only low levels of water. Preferably, the level of water is less than 20 wt % based on the weight of the smooth phase, more preferred less than 15 wt%, most preferred from 5 to 12 wt%. Most preferably the smooth phases are substantially free from water, which means that apart from low levels of moisture (e.g. for neutralisation or as crystal water) no additional added water is present.

**[0027]** Preferably the smooth phase is transparent or translucent. Preferably, this means that the composition has an optical transmissivity of at least 10%, most preferably 20%, still more preferably 30%, through a path length of 0.5 cm at 25° C. These measurements may be obtained using a Perkin Elmer UV/VIS Spectrometer Lambda 12 or a Brinkman PC801 Colorimeter at a wavelength of 520nm, using water as the 100% standard.

**[0028]** The transparency or translucency of the compositions according to the invention does not preclude the composition being coloured, e.g. by addition of a dye, provided that it does not detract substantially from clarity.

**[0029]** In an advantageous embodiment of the invention, the smooth phase comprises from 30-100 %wt of non-soap surfactants, more preferred 40 to 90 %wt (based on the total weight of said smooth phase), more preferred from 50 to 80 %wt. It has been found that the combination of a separate smooth first region and these high non-soap surfactant levels provide very good dispersing and cleaning properties to the tablet.

**[0030]** In another preferred embodiment of the invention, the detergent tablets comprise a first region (as described above) in combination with a smooth region. The smooth region advantageously comprises 50-100 %wt of non-soap surfactants for examples 60 to 90 %wt in combination with optional ingredients such as to 0 to 50 wt% soluble materials (as described above) 0 to 40 wt% diluent materials (as described above) and 0 to 20 wt% (as described above) of water.

**[0031]** The non-soap surfactants in said smooth phase may for example be anionic, nonionic or cationic non-soap surfactants or mixtures thereof. Relatively low levels of soap may also be present, for example up to 10 %wt based on said third smooth phase.

**[0032]** Preferably, the total weight of surfactants in the smooth phase is from 2 to 20 grammes, more preferred from 3 to 10 grammes.

Compacted third region

**[0033]** In a preferred embodiment of the invention, the tablet may be a multi-phase tablet wherein the phases other than the first phase (as described above) and the smooth phases as described above comprise no or only low levels of non-soap surfactants. Especially the third phase is a solid phase of compacted materials. Preferably the level of non-soap surfactants in said solid phase is less than 10 %wt (based on the total weight of said phase), more preferred from 0 to 9 %wt, most preferred from 1 to 8 %wt.

**[0034]** In a first embodiment of the invention, the detergent tablets comprise a first region (as described above) in combination with a second smooth region (as described above) and a third solid region, for example prepared by compression of a particulate composition.

**[0035]** Although the third 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 third region. Preferably, the bleach system is predominantly present in the third region. Preferably the enzymes are predominantly present in the third 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 third region, more preferred more than 98 %wt, most preferred substantially 100 %wt.

**[0036]** The above description of the tablet has been given with reference to a tablet constituted by two or three regions. It will however be understood that each of the regions may be composed of a limited number of discrete regions. Similarly, the smooth second region or the solid third region may be composed of a limited number (say 1-5) of parts e.g. separate layers in the tablet.

**[0037]** Detergent tablets according to the invention are preferably manufactured by a process involving the application of pressure to a particulate mixture. Advantageously, the preparation of the first phase may involve the dosing of a

particulate mixture comprising encapsulated perfume and particles of de-formable material optionally in combination with other materials as described above, followed by the exertion of moderate pressure, preferably above the yield stress of the particles.

**[0038]** Although applicants do not wish to be bound by any theory it is believed that the advantageous process of the invention works as follows: typically, compaction of soft solids lead to a decrease in inter-particle porosity relative to a particle bed simply due to reduced air entrapment. However, at a certain compaction force (yield stress) the individual particles will deform and flow into the inter particle voids leading to a semi-continuous network of intermeshed particles with no or low porosity and a smooth appearance.

**[0039]** A multi-phase tablet comprising a first phase (as described above) may advantageously be made by a process, comprising the steps of:

(a) inserting a first particulate composition into a tablet mould;

(b) inserting a second particulate composition into said tablet mould, wherein said first particulate composition comprises an encapsulated perfume and at least 50 %wt of deformable particles, wherein said particles comprise at least 10 %wt, preferably at least 25%wt, more preferably at least 50%wt, of a plastically deformable substance;

(c) compression of the particulate compositions to form a compressed tablet comprising discrete regions, wherein the first region is formed by said compressed first composition and the second region is formed by said compressed second composition.

**[0040]** Preferably, step (a) takes place before step (b). Preferably the first particulate composition is such that upon compression a solid phase of compressed particulate material is formed.

**[0041]** In a preferred embodiment of the invention, the first particulate composition is pre-compressed at a force of 0.1 to 20 kN/cm<sup>2</sup> between steps (a) and (b). In another preferred embodiment, the particulate composition is flattened between steps (a) and (b).

**[0042]** Preferably, the (co-) compression of the combination of the first and the solid region(s) takes place at a force of from 0.05 to 20 kN/cm<sup>2</sup>. Especially if the solid region has been pre-compressed the co-compression in step (c) can advantageously be at a force of 0.1- 10 kN/cm<sup>2</sup>, more preferred 0.5 to 5 kN/cm<sup>2</sup>. If the solid region has not been pre-compressed, the co-compression preferably takes place at a force of 0.5- 100 kN/cm<sup>2</sup>., more preferred 0.7-50 kN/cm<sup>2</sup>., most preferred 1-10 kN/cm<sup>2</sup>.

**[0043]** If the tablet of the invention comprises a first phase as described above, this phase may also be manufactured separately by compression of a particulate material e.g. at the compaction forces as indicated above.

**[0044]** Alternatively, the first phase may be prepared by other methods for example the spraying of a composition for example onto the (pre) compressed compacted tablet phase. Another suitable method for the preparation of a soap rich phase may involve casting or extrusion of a composition.

**[0045]** Optionally, the smooth phase may also be prepared e.g. by extrusion, casting or other shaping methods.

**[0046]** Separately prepared phases can then be adhered to other parts of the tablet for example by gentle pressing or by usage of an adhesive material.

**[0047]** Similarly, a separately prepared solid phase e.g. of compressed particulate materials can be combined with one or more pre-prepared phases e.g. by gentle co-compression.

**[0048]** A tablet of this invention may be intended for use in machine dishwashing. Such tablets will typically contain salts, such as over 60 wt% of the tablet.

**[0049]** 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.

**[0050]** Another preferred possibility is that a tablet of this invention will be intended for fabric washing. In this event the tablet will be likely to contain at least 2 wt%, probably at least 5 wt%, up to 40 or 50 wt% soap surfactant based on the whole tablet, and from 5 to 80 wt% detergency builder, based on the whole tablet.

**[0051]** Materials which may be used in tablets of this invention will now be discussed in more detail.

#### Surfactant Compounds

**[0052]** Compositions which are used in tablets of the invention will contain one or more detergent surfactants. In a fabric washing composition, these preferably provide from 5 to 50% by weight of the overall tablet composition, more preferably from 8 or 9% by weight of the overall composition up to 40% or 50% by weight. Surfactant may be anionic (soap or soap), cationic, zwitter-ionic, amphoteric, nonionic or a combination of these.

**[0053]** 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.

**[0054]** Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkyl-



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.

**[0067]** Conceivably, a water-insoluble detergency builder could be a layered sodium silicate as described in US 4664839.

**[0068]** NaSKS-6 is the trademark for a crystalline layered silicate marketed by Hoechst (commonly abbreviated as "SKS-6"). NaSKS-6 has the delta- $\text{Na}_2\text{SiO}_5$  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.

**[0069]** 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.

**[0070]** 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.

**[0071]** At least one region (preferably the second region) of a fabric washing tablet preferably include polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers which can function as builders and also inhibit unwanted deposition onto fabric from the wash liquor.

#### Bleach System

**[0072]** Tablets according to the invention may contain a bleach system in at least one region of a tablet, preferably in the second region. This preferably comprises one or more peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with activators to improve bleaching action at low wash temperatures. If any peroxygen compound is present, the amount is likely to lie in a range from 10 to 25% by weight of the composition.

**[0073]** 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.

**[0074]** 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

**[0075]** The detergent tablets of the invention may also contain (preferably in the second region) one of the detergency enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains. Suitable enzymes include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark. Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in amount of from about 0.1% to about 3.0% by weight of the composition; and these granules or marumes present no problems with respect to compaction to form a tablet.

**[0076]** The detergent tablets of the invention may also contain (preferably in the second region) a fluorescer (optical brightener), for example, Tinopal (Trade Mark) DMS or Tinopal CBS available from Ciba-Geigy AG, Basel, Switzerland. Tinopal DMS is disodium 4,4'-bis-(2-morpholino-4-anilino-s-triazin-6-ylamino) stilbene disulphonate; and Tinopal CBS is disodium 2,2'-bis-(phenyl-styryl) disulphonate.

**[0077]** An antifoam material is advantageously included (preferably in the second region), especially if a detergent tablet is primarily intended for use in front-loading drum-type automatic washing machines. Suitable antifoam materials are usually in granular form, such as those described in EP 266863A (Unilever). Such antifoam granules typically

comprise a mixture of silicone oil, petroleum jelly, hydrophobic silica and alkyl phosphate as antifoam active material, absorbed onto a porous absorbed water-soluble carbonate-based inorganic carrier material. Antifoam granules may be present in an amount up to 5% by weight of the composition.

**[0078]** It may also be desirable that a detergent tablet of the invention includes an amount of an alkali metal silicate, particularly sodium ortho-, meta- or disilicate. The presence of such alkali metal silicates at levels, for example, of 0.1 to 10 wt%, may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some measure of building and giving processing benefits in manufacture of the particulate material which is compacted into tablets.

**[0079]** A tablet for fabric washing will generally not contain more than 15 wt% silicate. A tablet for machine dishwashing will often contain more than 20 wt% silicate. Preferably, the silicate is present in the second region of the tablet.

**[0080]** Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the second region) include anti-redeposition agents such as sodium carboxymethylcellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose, fabric-softening agents; heavy metal sequestrants such as EDTA; perfumes; and colorants or coloured speckles.

**[0081]** Further ingredients which can optionally be used in tablets of the invention, preferably in the second region, are dispersing aids. Examples of suitable dispersing aids are water-swelling polymers (e.g. SCMC) highly soluble materials (e.g. sodium citrate, potassium carbonate or sodium acetate) or sodium triphosphate with preferably at least 40% of the anhydrous phase I form.

## Particle Size and Distribution

**[0082]** The first soap rich region of the detergent tablet may advantageously be prepared by compacting particles with a high soap content as described above. Preferably these particles have a mean particle size of from 100 to 1000  $\mu\text{m}$ . The second region of a detergent tablet of this invention, is a preferably a matrix of compacted particles.

**[0083]** Preferably, the particulate composition has a mean particle size in the range from 200 to 2000  $\mu\text{m}$ , more preferably from 250 to 1400  $\mu\text{m}$ . Fine particles, smaller than 180  $\mu\text{m}$  or 200  $\mu\text{m}$  may be eliminated by sieving before tableting, if desired, although we have observed that this is not always essential.

**[0084]** While the starting particulate composition may in principle have any bulk density, the present invention is especially relevant to tablets made by compacting powders of relatively high bulk density, because of their greater tendency to exhibit disintegration and dispersion problems.

**[0085]** Such tablets have the advantage that, as compared with a tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

**[0086]** 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.

**[0087]** Tableting machinery able to carry out the manufacture of tablets of the invention is known, for example suitable tablet presses are available from Fette and from Korch.

**[0088]** 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.

**[0089]** 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.

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

### Example 1

**[0090]** Preparation of a detergent tablet comprising a first phase comprising encapsulated perfumes, a second smooth phase and a third solid compacted phase.

Preparation of perfume granules:

**[0091]** Perfume encapsulates were made with the composition below using a two step high shear mixer- fluidised bed process using 34.8 weight parts of zeolite 4A and 17.5 weight parts of sugar onto which 17.7 weight parts of a slurry of perfume capsules (slurry containing 42wt% of melamine formaldehyde-urea capsules incorporating perfume as obtained

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from Basf) is applied by spraying with a nozzle.

Spray system: Eminent E13 spray gun

Nozzle spec: 20 HTE nozzle.

Mixer: standard concrete mixer.

Mixing time: 15 minutes.

Batch size: 20kg.

Spray-on rate: 0.8 kg hr<sup>-1</sup>

Capsule concentration in slurry: 42wt% (balance: water)

### Solid compressed phase

**[0092]** A solid compacted -third- phase was prepared as follows.

A powder was made of the following composition(I) by pre-granulating the granule ingredients, followed by post-dosing the rest of the ingredients.

Composition I:

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

**[0093]** 20 grammes of the particulate composition are inserted into a 45 mm die of a tableting machine, optionally followed by a flattening step. The whole material is compressed at 30kN into a single tablet.

Deformable -first-phase preparation:

**[0094]** 1 part by weight of C12-C14 soap particles was mixed with 1 weight part of sodium sulphate and 0.6 part of the perfume granule. 2 grammes of the mixture were dosed in the tablet mould on top of the solid phase, followed by a second compression step of 10 kN resulting in the formation of a first perfume rich phase on top of the solid phase.

Smooth second phase preparation:

**[0095]** The smooth phase was prepared of the following composition:

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

**[0096]** The mixture was heated to 80°C and cast into moulds and cooled to 20°C to form firm, 5 grams smooth, parts

of 45mm diameter.

[0097] A smooth part is then applied to the top of the tablet e.g. by gentle compression. The resulting tablet is a three layer tablet whereby the perfume rich layer is located as a barrier between the smooth phase and the solid compacted phase.

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Example 2

[0098] A two-layer reference tablet A was prepared as follows:

1. 33 grams of composition I was inserted into a 45 mm tableting die.
2. 1 weight part of composition I was mixed with 0.1 parts of perfume granules of example 1, then 6.1 grams of the mixture were inserted into the die on top on top of the layer of composition I.
3. The compositions were co-compressed at 19 kN to result in reference tablet (A) comprising a perfume rich phase on top of a compacted solid phase.

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[0099] A two layer tablet according to the invention B was prepared as follows:

1. 33 grams of composition I was inserted into a 45 mm tableting die.
2. One weight part of composition I was mixed with 1 weight part of plastically deformable C12-C14 soap particles and 0.2 parts of perfume granules of example 1 to form a mixture.
3. 6.1 grams of the mixture were inserted into the die on top of the layer of composition I.
4. The compositions were co-compressed at 19 kN to result in a cleaning tablet (B) comprising a perfume rich phase on top of a compacted solid phase.

[0100] After formation of the reference tablet A and the cleaning tablet B, both tablets were assessed to determine the extent to which the perfume encapsulates were broken during the production process. This was determined by checking the leakage of perfume from the tablet by quietly dissolving the tablet in water at 40 °C and measuring the headspace above the tablets after 30 minutes. The higher the perfume concentration in the headspace, the greater the number of capsules that were broken during tableting. The leakage was calculated as a % of what the leakage would have been had all the encapsulates suffered damage.

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[0101] The following results were obtained:

Tablet A: 15% leakage before tableting 50% leakage after tableting.

Tablet B: 15% leakage before tableting 32% leakage after tableting.

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[0102] This example shows that the incorporation of the perfume encapsulates embedded in a matrix of a material which is plastically deformable (here soap) results in a markedly lower degree of damage to the perfume encapsulates during the compression step.

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**Claims**

1. A cleaning tablet which has a plurality of discrete regions with differing compositions, **characterised in that** at least a first region of the tablet comprises an encapsulated perfume material embedded in a matrix of a material which is plastically deformable under pressure.

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2. A cleaning tablet according to claim 1 or 2, wherein said first region comprises

- (a) from 10-90 wt% of plastically deformable substance;
- (b) from 0 to 80 wt% of water-soluble solid materials or mixtures;
- (c) from 1 to 20 wt% of encapsulated perfume particles.

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3. A cleaning tablet according to one or more of the preceding claims, wherein the encapsulated perfume materials are core in shell encapsulates.

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4. A cleaning tablet according to one or more of the preceding claims, wherein the plastically deformable substance is selected from polyethylene glycols, soap, long chain nonionic surfactants, primary alkyl sulphates, linear alkyl benzene sulphonates or mixtures thereof which are solid at ambient temperature.

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5. A cleaning tablet according to one or more of the preceding claims, wherein the water-soluble solid material is selected from sodium and potassium citrates, chlorides, acetates, sulphates, carbonates, percarbonates or bicarbonates; urea and sugar or mixtures thereof.
- 5 6. A cleaning tablet according to one or more of the preceding claims, additionally comprising a second region which is a smooth region.
7. A cleaning tablet according to one or more of the preceding claims, additionally comprising a region of compacted particulate materials.
- 10 8. A cleaning tablet according to claim 1, additionally comprising a second smooth region and a third solid region of compacted particulate materials, wherein the first region is present as a barrier between the smooth region and the solid region.
- 15 9. A method of producing a cleaning tablet according to one or more of the preceding claims, whereby the first region is obtained by compressing a particulate material comprising at least 10 wt% of plastically de-formable substance and from 1 to 20wt% of perfume encapsulates.
- 20 10. A method according to claim 9, wherein the compression takes place at a pressure from 0.1 to 2 kN/cm<sup>2</sup>.

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The present search report has been drawn up for all claims

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EPO FORM 1503 03.82 (P04C01)

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