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(54) **Cleaning compositions**

(57) A detergent tablet wherein the tablet comprises a first phase and a second phase, wherein said first phase comprises a compacted particulate material and wherein said first phase comprises phosphate builder having an average water content of from 5.5 to 22.7 wt%

and wherein at least 40 wt% of said phosphate builder in said first phase is in Phase I form, and wherein said second phase is a smooth phase.

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**Description**

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

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

**[0003]** It is desirable that detergent tablets have sufficient strength to keep them intact during storage and handling prior to use but also disintegrate rapidly when in contact with a limited amount of water.

**[0004]** Strength when dry and rapid disintegration when wet are desirable properties which militate against each other. When tablets are made by compacting a composition in particulate form, the force used for compaction affects these two properties.

**[0005]** Tablets formed using a low compaction pressure tend to crumble and disintegrate on handling and packing; while more forcefully compacted tablets may be sufficiently cohesive but can be slow to disintegrate or disperse to an adequate extent in use.

**[0006]** It has been described in EP 716 144 to use an organic polymeric binder material to enhance the strength of detergent tablets. However the use of such binder materials may undesirably increase the costs and also may lead to a more complicated process for manufacturing the tablets.

**[0007]** Also it has been suggested that if a tablet contains organic surfactant, this can function as a binder, plasticising the tablet. However the presence of the surfactants may sometimes retard disintegration of the tablet by forming a viscous gel when the tablet comes into contact with water. Thus, the presence of surfactant can make it more difficult to achieve good disintegration while maintaining adequate strength.

**[0008]** The object of the present invention is to provide detergent tablets comprising one or more phases of compacted particulate material and one or more smooth phases wherein said phase(s) does not require very high compaction pressures to ensure adequate strength for the tablet, and wherein the use of binder materials can be avoided.

**[0009]** It has now been found that a detergent tablet wherein the tablet comprise a first phase composed of a compacted particulate material, wherein said first phase comprises phosphate builder having a water content of from 5.5 to 22.7 wt% (based on the weight of the phosphate builder) and wherein at least 40% of said phosphate builder is in Phase I form, and a second phase which is a smooth phase, provides good dispersing and cleaning properties and said detergent tablet has a good strength.

**[0010]** Accordingly in a first aspect the present invention relates to a detergent tablet wherein the tablet comprises a first phase and a second phase, wherein said first phase comprises a compacted particulate material and wherein said first phase comprises phosphate builder having an average water content of from 5.5 to 22.7 wt% and wherein at least 40 wt% of said phosphate builder in said first phase is in Phase I form, and wherein said second phase is a smooth phase.

**[0011]** Some prior disclosures describe the use phosphate builders in cleaning tablets, for example EP 839 906 discloses the use in tablets of sodium tripolyphosphate containing between 1.0 and 5.0 wt% of water of hydration. WO 00/22090 discloses a anti-calcium tablets comprising a phosphate builder with 6 to 16 wt% of water. WO 02/42398 discloses detergent tablets comprising sodium tripolyphosphate which may comprise hydration levels of from 0.5 to 10 wt%. WO 00/32741 discloses detergent tablets comprising partially hydrated sodium tripolyphosphate.

**[0012]** EP 1239028 describes the production of multi-layer detergent tablets wherein partially hydrated sodium tripolyphosphate having up to 5% of water of hydration.

**[0013]** WO 00/12671 discloses a detergent tablet with a sodium tripolyphosphate builder which is a mixture of sodium tripolyphosphate hexahydrate, 1-18% Phase I anhydrous sodium tripolyphosphate and Phase II anhydrous sodium tripolyphosphate.

**[0014]** Preferably tablets of the invention are of cylindrical shape wherein the two main surfaces (upper side and bottom side) are substantially flat.

**[0015]** As indicated above, the invention relates to a multiphase tablet wherein a compacted particulate phase and a smooth phase are present and optionally one or more other smooth or compacted particulate phase(s) are present. Especially preferably tablets of the inventions comprise a limited number of compacted phases (up to 5, preferably one compacted phase) in combination with a limited number of smooth phases (up to 5, preferably one smooth phase).

**[0016]** The regions of a multi-phase tablet are possibly separate layers within a tablet. However, a discrete region of a tablet could also have other forms for example one or more core(s) or insert(s). In a preferred embodiment the first region is a layer of compacted particulate material and the second region is a smooth layer. In a further advantageous embodiment the first region is a core or insert of e.g. a smooth material embedded in the second region which is for example a layer of compacted particulate material.

**[0017]** The multi-phase tablet comprises a compacted phase preferably having a weight of from 2 to 50 grammes, more preferred from 3 to 40 grammes. Preferably the other phases together including one or more smooth phases have a weight of 10 to 50 grammes, more preferred 15 to 40 grammes, whereby each phase has a weight of from 2

to 50 grammes, more preferred from 3 to 40 grammes.

**[0018]** In a preferred embodiment of the invention the compacted particulate phase as described above comprises no or only low levels of surfactants. Preferably the level of surfactants in the compacted particulate 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%. It has been found that the absence of high levels of surfactants -which normally would act as a binder- in the compacted particulate phase does not lead to unacceptable low coherence between the particulates, provided the compacted particulate phase comprises a phosphate builder having a water content between 5.5 and 22.7 % based on the weight of the phosphate builder.

**[0019]** Preferably the level of phosphate builder having a water content between 5.5 and 22.7 % in the compacted particulate phase is from 10 to 90 wt%, more preferred from 15 to 80 wt%, most preferred from 20 to 70 wt% based on the total weight of the compacted phase. If desired a mixture of phosphate builders may be used, provided that the average degree of hydration of the phosphate builder is at within the ranges as described herein. Preferably the phosphate builder is STP. Generally the moisture in the phosphate builder will be present as crystal water. The upper limit for the moisture level of the phosphate builder is 22.7 wt%, referring to the fully hydrated STP hexahydrate state. Preferably the average water content is at least 7.5 wt% (based on the total amount of phosphate present in the compacted phase), more preferred more than 10 wt% preferably up to 20 wt%.

**[0020]** Preferably the phosphate builder is for at least 50% by weight in the Phase I form.

**[0021]** In a preferred embodiment of the invention the detergent tablets comprise a silicate. Especially preferable the level of silicate (preferably in the form of alkali metal silicate) is from 1 to 15 wt% based on the weight of the phosphate builder, more preferred from 2 to 12 wt%, most preferred from 4 to 8 wt%. Preferably the silicate is present in the same phase of the tablet as the phosphate builder.

**[0022]** Although the compacted particulate region may comprise some 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 compacted particulate region (s). Also preferably the bleach system is predominantly present in the compacted particulate region. Also preferably the enzymes are predominantly present in the compacted particulate 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 compacted particulate region, more preferred more than 98 wt%, most preferred substantially 100 wt%.

**[0023]** The invention relates to the combination of at least one compacted particulate phase and at least one smooth phase. 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.

**[0024]** In an advantageous embodiment of the invention the smooth phase comprises from 20-80 wt% of non-soap surfactants (based on the total weight of said smooth phase), more preferred from 25 to 75 wt%, most preferred 30 to 70 wt%. It has been found that the combination of a separate smooth second region and these high surfactant levels provide very good dispersing and cleaning properties to the tablet.

**[0025]** Preferably the non-soap surfactants in the smooth region comprise a combination of anionic surfactants and 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 part.

**[0026]** The smooth region may also contain structuring materials which leads on the one hand to a desired firm consistency of the smooth phase but on the other hand retains the smooth nature of the phase. Examples of such structuring materials are for example soap, urea, acetate, sugar etc. Preferably the level of structurant materials is from 0.5 to 40 wt% based on the total weight of the smooth phase.

**[0027]** The smooth region of the tablet may also contain diluent materials for example polyethyleneglycol, dipropyl-ene glycol, isopropanol or (mono-)propyleneglycol. Preferable the level of these diluents is from 0 to 40 wt%, more preferred 1 to 20, most preferred from 4 to 15 wt% based on the weight of the smooth phase.

**[0028]** 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.

**[0029]** Preferably the total weight of surfactants in the smooth phase is from 2 to 20 grammes, more preferred from 3 to 10 grammes.

**[0030]** Preferably the smooth phase comprises no or only low levels of ingredients such as builders, bleach activators and bleach materials. Preferably the level of these ingredients in the smooth phase is less than 5 wt%.

**[0031]** 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. In addition to the regions described above, tablets of the invention may optionally comprise further regions, for example the tablet may be partially or wholly coated.

**[0032]** The compacted particulate phase of the cleaning tablets according to the invention is preferably manufactured by introducing a particulate material into a tablet mould followed by compaction. Generally compaction pressures will be typically be in the range of from 0.05 to 5 kN/cm<sup>2</sup>. Due to the fact that the hydrated phosphate builder acts as a binder, generally the compaction pressure may be relatively low. If the cleaning tablet of the invention comprises more than one compacted particulate phase, this may be prepared by consecutive compression steps such as for example described in EP 1239028. The smooth phases could for example be prepared by heating of the ingredients followed by cooling, casting and/or by extrusion. The smooth parts can then for example be adhered to the compacted phase by co-compression.

**[0033]** Preferably the (co-)compression of the combination of the smooth and the compacted 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 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>. Such co-compression generally leads to good adherence of the first region to the second region and avoids the need of applying an adhesive material between the smooth and solid region. Another advantage of the method of the invention is that it can be carried out in a normal tablet press without the need of adaptation of the shape of the pressing surfaces.

**[0034]** A tablet of this invention may be intended for use in machine dishwashing. Such a tablet is likely to contain surfactant in a low concentration such as 0.5 to 2 wt% based on the whole tablet, although higher concentrations ranging up to 10 wt% may be used. Such will typically contain salts, such as over 60 wt%, often over 85 wt% of the tablet.

**[0035]** 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.

**[0036]** Another preferred possibility is that a tablet of this invention will be intended for fabric washing. In this event the tablet will be likely to contain at least 2 wt%, probably at least 5 wt%, up to 40 or 50 wt% non-soap surfactant based on the whole tablet, and from 5 to 80 wt% detergency builder, based on the whole tablet.

**[0037]** Materials which may be used in tablets of this invention will now be discussed in more detail.

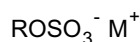
#### Surfactant Compounds

**[0038]** 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.

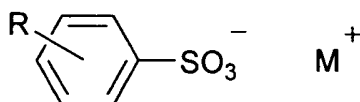
**[0039]** Anionic surfactant may be present in an amount from 0.5 to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the tablet composition.

**[0040]** 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<sub>8</sub>-C<sub>15</sub>; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

**[0041]** 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<sup>+</sup> is a solubilising cation, is commercially significant as an anionic surfactant.



**[0042]** Linear alkyl benzene sulphonate of the formula

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

**[0043]** 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.

**[0044]** 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.

**[0045]** Soaps for use in accordance to the invention are preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from beef tallow.

**[0046]** 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.

**[0047]** Specific nonionic surfactant compounds are alkyl (C<sub>8-22</sub>) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C<sub>8-20</sub> 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.

**[0048]** Especially preferred are the primary and secondary alcohol ethoxylates, especially the C<sub>9-11</sub> and C<sub>12-15</sub> primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

**[0049]** 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.

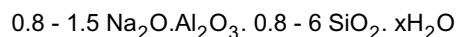
**[0050]** Many nonionic surfactants are liquids. These may be absorbed onto particles of the composition.

**[0051]** 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

**[0052]** A composition which is used in tablets of the invention will usually contain from 5 to 80%, more usually 15 to 60% by weight of detergency builder. This may be provided wholly by water soluble materials (such as the phosphate builder with the hydration content of more than 5.5 wt%), but may also in part be 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.

**[0053]** Alkali metal aluminosilicates are sometimes 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:



**[0054]** These materials contain some bound water (indicated as "xH<sub>2</sub>O") 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<sub>2</sub> 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.

**[0055]** 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.

**[0056]** 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<sub>2</sub>SiO<sub>5</sub> 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<sub>x</sub>O<sub>2x+1</sub>·yH<sub>2</sub>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.

**[0057]** 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. As described above the average moisture content of the phosphate builder present in the compacted particulate phase(s) is at least 5.5 wt%, more preferred more than 7.5 wt% most preferred more than 10 wt% up to 20 wt% or even total hydration (22.7 wt% for STP hexa hydrate).

**[0058]** 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.

**[0059]** At least one region (preferably the compacted 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.

#### 5 Bleach System

**[0060]** Tablets according to the invention may contain a bleach system in at least one region of a tablet, preferably in the compacted particulate phase. This bleach system 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.

**[0061]** 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, tetraacetylene 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.

**[0062]** 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.

#### 25 Other Detergent Ingredients

**[0063]** The detergent tablets of the invention may also contain (preferably in the region of the compacted particulate phase) 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.

**[0064]** The detergent tablets of the invention may also contain (preferably in the region of the compacted particulate phase) 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.

**[0065]** An antifoam material is advantageously included (preferably in the region of the compacted particulate phase), 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.

**[0066]** 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.

**[0067]** A tablet for fabric washing will generally not contain 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 region of the compacted particulate phase of the tablet.

**[0068]** Further ingredients which can optionally be employed in a region of a fabric washing detergent of the invention tablet (preferably the region of the compacted particulate phase) 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.

**[0069]** 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-swellaible 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.

5 Particle Size and Distribution

[0070] The second region of a detergent tablet of this invention, is a preferably a matrix of compacted particles.

[0071] 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  
10 tableting, if desired, although we have observed that this is not always essential.

[0072] While the starting particulate composition may in principle have any bulk density, the present invention is especially relevant to tablets made by compacting powders of relatively high bulk density, because of their greater tendency to exhibit disintegration and dispersion problems. Such tablets have the advantage that, as compared with a tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

[0073] 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.

[0074] 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.

[0075] 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.

[0076] 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.

[0077] The invention will further be illustrated in the following examples:

30 Note: STP used in all examples was 70% in Phase I form

I.-Preparation of the Compacted Particulate Phase(s)

35 Example 1

[0078] Detergent tablets were manufactured by introducing 21 grammes of a particulate composition into a tablet mould having an diameter of 45 mm. Composition A (comparative example) comprised a STP phosphate builder with a hydration water content of 3.5 wt%. Composition B comprised a mixture of STP having a hydration water content of 3.5 wt% (Polypray H) and STP having a hydration water content of 13.4 wt%. The high moisture STP is prepared by  
40 spraydrying Polypray H with water followed by drying to the moisture content indicated.

[0079] Both mixtures were compressed until tablets of a tablets strength (Fmax) of about 95 N were obtained. Formulation A required the use of a compression pressure of about 32 kN to achieve the desired strength. Formulation B only required a compression pressure of about 6 kN. The higher compression pressure for the comparative tablets leads to a lower porosity and a lower rate of dissolution/dispersion for composition A as compared to composition B  
45 according to the invention.

Ingredient	Composition A; parts by weight	Composition B; parts by weight
STP (Polypray H; 3.5% moisture)	60	27
STP (L2209; moisture 13.4 wt%)	-	33
Antifoam granule	3.5	3.5
Fluorescer adjunct	3	3
Sodium disilicate	5	5
TAED (83%)	5	5
Sodium percarbonate	21	21

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(continued)

Ingredient	Composition A; parts by weight	Composition B; parts by weight
EDTMP	2.5	2.5

### Example 2:

**[0080]** Example I was repeated by replacing the 60 parts by weight of STP by:

Composition C: a 75/25 mixture of Polypray H (3.5% moisture) and fully hydrated STP (22.7 % moisture)

Composition D: a 50/50 mixture of Polypray H (3.5% moisture) and fully hydrated STP (22.7 % moisture)

Composition A (as in example I) required a compression force of about 61 kN in order to obtain an Fmax of about 50 N. Composition C required a compression force of about 48 kN in order to obtain an Fmax of about 50N.

Composition D required a compression force of about 40 kN to obtain an Fmax of about 50 N.

### Example 3

**[0081]** Example I was repeated by replacing the 60 parts by weight of STP by:

Composition E: a 75/25 mixture of 75 parts Polypray H (3.5% moisture) and 25 parts of fully hydrated STP (22.7 % moisture) comprising 6wt% on phosphate of sodium silicate.

Composition F: a 50/50 mixture of 75 parts Polypray H (3.5% moisture) and 25 parts of fully hydrated STP (22.7 % moisture) comprising 6wt% on phosphate of sodium silicate.

Composition A (as in example I) required a compression force of about 72 kN in order to obtain an Fmax of about 61 N. Composition E required a compression force of about 47 kN in order to obtain an Fmax of about 61 N.

Composition F required a compression force of about 33 kN to obtain an Fmax of about 61 N.

### II.- Preparation of smooth phase(s)

#### Example I

**[0082]** 7 kg of anionic surfactant (Dobanic acid 103 ex Shell) and 6kg of nonionic surfactant (Lutensol AO7 ex BASF) were mixed and neutralised to a pH of 9 using a 50% NaOH solution.

**[0083]** 10 wt% (based on the weight of neutralised blend) of soap fatty acid (Pristerene 4916 fatty acid ex Uniqema) was added. 5 to 10 wt% (based on the weight of neutralised blend) dipropylene glycol (ex Vopak) was also added to the mixture. The mixture was further neutralised with a 50% NaOH solution to a pH of 11.

**[0084]** After neutralisation to pH of 11, the mixture was pumped into a sequence of 2 stainless steel tubes by a Maag Sinox P7 pump or a piston pump, type SIBa HK 05016SST4000M000 ex Prominent, Vleuten (NL). Both tubes were double jacketed. The first tube was 2.5m long and had an inner diameter of 73mm. The second tube was 1.5m long and had an inner diameter of 45mm. The tubes were connected by a 10cm long pipe.

**[0085]** The extrusion was performed in the absence of a die-head.

**[0086]** The mixture was pumped into the tubes at a temperature of 85°C at a throughput of 4 kg/hr. The first tube was cooled using a water bath at 40°C. The second tube was cooled using a 50:50 weight mixture of ethylene glycol and water. The coolant temperature was -15°C. The material coming out of the second tube had a temperature of about 20 C and was collected and divided into bars of around 0.5m.

**[0087]** After storage the bars were cut into slices of 5 gramme each.

### III.- Preparation of Multiphase tablets

#### Example 1:

**[0088]** A detergent powder was made of the following composition by pregranulating the granule ingredients, followed by post-dosing the rest of the ingredients

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<i>Ingredient</i>	<i>Parts by weight</i>
<b>Granules</b>	
Na-las	1.1
Nonionic 7EO	0.5
C12 soap	0.1
NaAc.3aq	0.3
Zeolite A24	2.4
Light soda ash	0.4
Moisture/minors	0.4
<b>Post-dose</b>	
EAG (17% silicone)	3.0
Fluorescer (15%)	2.2
STP *	60.0
Na-disilicate (80%)	3.8
TAED (83%)	4.3
Percarbonate	16.9
Dequest 2047	1.9
Minors/ enzymes/colour	to 100
Note: STP as in composition B described above.	

**[0089]** Smooth parts of 5 gramme were prepared as above.

The tablets were made as follows: 20 grammes of the powder are inserted into a 45 mm die of a tableting machine, optionally followed by a flattening step, followed by addition of a single smooth part on top of the powder bed. After addition of the smooth onto the powder bed or flattened powder, the whole material is compressed at 30kN into a single tablet, followed by ejection of the tablet. This results in a tablet with a smooth part embedded in the cleaning tablet. The density of the powdered region is 1.5 kg/litre, the density of the smooth part is 1.0 kg/litre. The height of the smooth part after compression is 3.4 mm, of the powdered part 11 mm.

**Claims**

1. A detergent tablet wherein the tablet comprises a first phase and a second phase, wherein said first phase comprises a compacted particulate material and wherein said first phase comprises phosphate builder having an average water content of from 5.5 to 22.7 wt% and wherein at least 40 wt% of said phosphate builder in said first phase is in Phase I form, and wherein said second phase is a smooth phase.
2. A detergent tablet according to claim 1 wherein the tablet comprises 2 to 5 phases, each phase having a weight of from 2 to 50 grammes, more preferred from 3 to 40 grammes.
3. A detergent tablet according to one or more of the preceding claims wherein the phosphate builder has an average water content of from 7.5 to 20 wt%, more preferred from 10.0 to 20.0 wt%.
4. A detergent tablet according to one or more of the preceding claims wherein the phosphate builder is for at least 50 wt% in the Phase I form.
5. A detergent tablet according to one or more of the preceding claims wherein the level of phosphate builder having an average water content from 5.5 to 22.7 wt% in said phase of compacted particulate material is from 10 to 90 wt%, more preferred from 15 to 80 wt%, most preferred from 20 to 70 wt%.
6. A detergent tablet according to one or more of the preceding claims comprising a silicate in an amount of 1 to 15 wt%, more preferred 2 to 12 wt%, most preferred 4 to 8 wt% of the phosphate builder.

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7. A detergent tablet according to one or more of the preceding claims wherein the phosphate builder is partially or fully hydrated STP.

5 8. A detergent tablet according to one or more of the preceding claims comprising a bleach material which is predominantly present in said phase of compacted particulate material.

9. A detergent tablet according to one or more of the preceding claims wherein the surfactant level in said phase of compacted particulate material is less than 10 wt%, more preferred from 0 to 9 wt%, most preferred from 1 to 8 wt%.

10 10. A detergent tablet according to one or more of the preceding claims, wherein the surfactant level in the smooth phase is from 20 to 80 wt%.

15 11. A method of preparing a detergent tablet according to one or more of the preceding claims comprising the step of compacting a particulate mixture at a pressure of 0.05 to 5 kN/cm<sup>2</sup>.

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EUROPEAN SEARCH REPORT

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