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(54) DETERGENT COMPOSITIONS IN TABLET FORM

REINIGUNGSMITTELZUSAMMENSETZUNGEN IN TABLETTENFORM

COMPOSITIONS DETERGENTES SOUS FORME DE COMPRIMES

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Description

[0001] This invention relates to detergent compositions in the form of tablets, especially tablets for use in fabric washing. Such tablets have the advantage that they do not require the user to measure out a volume of powder or liquid. Instead one or several tablets provide an appropriate quantity of composition for washing a single load in a washing machine or possibly by hand. They are thus easier for the consumer to handle and dispense.

5 [0002] Detergent compositions in tablet form have been described in a number of documents and are sold commercially.

[0003] Such tablets are generally made by compressing or compacting a quantity of detergent composition in particulate form. It is desirable that tablets should have adequate mechanical strength when dry before use, yet disintegrate and disperse/dissolve quickly when added to wash water. There is difficulty in achieving both properties simultaneously. As more pressure is used when a tablet is compacted, so the tablet density and strength rise, but there is also a reduction in the speed of disintegration/dissolution when the tablet comes into contact with wash water at the time of use. Organic detergent serves as a binder, but a typical quantity of such detergent can also retard disintegration and dissolution of a tablet. Our EP-A-466485 explains that as a tablet is wetted, anionic detergent can form viscous phases which retard penetration of water into the tablet interior.

10 [0004] This EP-A-466485 describes detergent tablets in which anionic detergent is contained within a first particulate component of the composition. This first particulate component provides from 2 to 40% of the overall composition. In most examples in this document the nonionic detergent was mixed with or carried in particles which provided a majority of the overall composition. Detergent compositions and tablets comprising nonionic surfactant as a minor proportion of 15 the particles in which the surfactant is present in are also disclosed in EP 716 144, EP 711 827, EP 839 906, EP 838 519 and WO 98/42817.

20 [0005] In some tablets which are currently marketed commercially, the anionic and nonionic detergent are jointly incorporated into a base powder which is mixed with other ingredients to form the composition stamped into tablets. The spray-dried base powder constitutes about 40% of the composition. It contains anionic detergent as approximately 25% 25 of the base powder and nonionic detergent as approximately 12% of the same powder.

[0006] In the present invention, detergent tablets are made using particles which incorporate a higher proportion of 30 nonionic detergent. According to the present invention, there is provided a detergent tablet according to claim 1.

[0007] These tablets may be either homogeneous or heterogeneous. In the present specification, the term "homogeneous" is used to mean a tablet produced by compaction of a single particulate composition, but does not imply that all 35 the particles of that composition will necessarily be of identical composition. The term "heterogeneous" is used to mean a tablet consisting of a plurality of discrete regions, for example layers, inserts or coatings, each derived by compaction from a particulate composition. In a heterogenous tablet, each discrete region of the tablet will preferably constitute at least 10% of the overall weight of the tablet.

[0008] Using such particles, we have been able to make tablets with a good combination of properties, notably strength 35 prior to use, and rapid disintegration when placed in contact with water at the time of use.

[0009] The particles may contain enough nonionic detergent to constitute at least 50% of their own weight. Preferably the composition of the tablet or region contains from 1 to 40 % by weight of nonionic detergent, at least half of which is 40 present as said particles.

[0010] Anionic detergent will frequently also be present and may be provided as particles which contain at least 20% 45 of their own weight of non-soap anionic detergent.

[0011] A tablet of this invention intended for fabric washing will generally contain, overall,

- at least 5%, better at least 8%, up to not over 40%, possibly not over 30%, by weight of non-soap organic detergent which is preferably a combination of anionic and nonionic detergents;
- at least 15%, better at least 20 or 25%, up to 80%, possibly not over 70 or 60% by weight of one or more detergency builders which may be water-soluble, water-insoluble or a mixture of soluble and insoluble builders;
- optionally other ingredients which may amount to at least 10% by weight of the tablet.

50 [0012] The invention could also be embodied as tablets for machine dishwashing, with a small percentage of nonionic detergent present such as 1 to 8% by weight, provided the tablet comprises said particles containing at least 45% nonionic detergent, from 20 to 99% detergency builder, and possibly no anionic detergent at all.

[0013] Constituent materials for detergent tablets will now be discussed in more detail, and various optional and 55 preferred features will be mentioned.

Nonionic Detergent Particles

[0014] The nonionic detergent particles used in this invention contain at least 45%, better at least 50% of their own weight of nonionic detergent (that is one or more organic compounds which are nonionic and have deterotive surfactant properties). Preferably they contain less than 10% by weight of synthetic (i.e. non-soap) anionic detergent, and preferably substantially no non-soap anionic detergent.

[0015] The nonionic detergent is primary or secondary alcohol ethoxylates, especially the C₉₋₁₁ and C₁₂₋₁₅ primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.

[0016] The nonionic detergent particles contain an inorganic carrier material which is water-insoluble. The insoluble carrier material may comprise silica or aluminosilicate, such as zeolite. However, it is preferred that, if aluminosilicate is present, the quantity is less than 10% by weight of the particles. The quantity of nonionic detergent may exceed 50% by weight of the particle, e.g. 52% or above.

[0017] Particles containing nonionic detergent absorbed on a solid carrier material can be made by spraying the nonionic detergent onto the carrier material in a granulator or some other type of mixing apparatus.

[0018] Other materials, serving to improve the physical properties of the particles, may also be included. Such materials are frequently referred to as "structuring agents". Examples are polyethylenepolypropylene glycol of average molecular weight in the region 4,000-12,000, sodium soap, polyvinyl alcohol of average molecular weight in the range 30,000-200,000, alkaline metal succinate etc may be present. The preferred quantity of structuring agent is in the region from 0.5 to 20% by weight. A structuring agent may be added with other ingredients or during a second step of granulation.

[0019] A preferred carrier is silica having an oil absorption capacity of at least 1.0 ml/g. Oil absorption capacity is a parameter which is well known and can be measured by the technique described in DIN ISO 787/5. Preferably, the oil absorption capacity is at least 1.5 ml/g, more preferably at least 2.0 ml/g.

[0020] Preferably, there is at least 10%, more preferably at least 15% of such silica in the particles, and the quantity of silica in the particles is greater than the quantity if any, of aluminosilicate. The particles may contain less than 10% of their own weight of aluminosilicate.

[0021] Nonionic detergent particles can be manufactured in a one step or two step process by mixing together components in a granulator (for example an Eirich RV02 granulator, or in equipment such as the Fukae mixer from Fukae Powtech Co of Japan, the Diosna V-series supplied by Dierks & Sohne Germany, the Pharma Matrix ex TH Fielder Ltd England, the Lodige CB series and the Dais T160 series from Dais Werke GmbH, Mannheim, Germany).

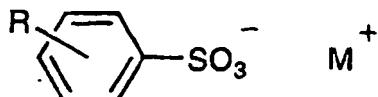
[0022] Nonionic detergent particles preferably have mean particle size in a range from 200 to 2,000 μ m such that at least 80% of these particles have a particle size in the range from 180 to 2,000 μ m. Nonionic detergent particles may provide from 1, better 2 or 3% up to 30% or possibly more of a tablet or a region of a tablet. More especially, such particles may constitute from 8 to 20% of a tablet or region of a tablet.

[0023] Generally a tablet or region of a tablet in accordance with this invention will contain from 1 % preferably from 3 to 40% by weight of nonionic detergent, at least half of which is present as said particles containing at least 40% of their own weight of nonionic detergent.

Anionic Detergent

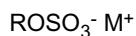
[0024] A tablet will frequently contain anionic detergent which is one or more non-soap organic compounds with deterotive surfactant properties.

[0025] The anionic detergent may comprise, wholly or predominantly, linear alkyl benzene sulphonate of the formula



50 where R is linear alkyl of 8 to 15 carbon atoms and M⁺ is a solubilising cation, especially sodium.

[0026] Primary alkyl sulphate having the formula



in which R is an alkyl or alkenyl chain of 8 to 18 carbon atoms especially 10 to 14 carbon atoms and M⁺ is a solubilising cation, is also commercially significant as an anionic detergent and may be used in this invention.

[0027] Frequently, such linear alkyl benzene sulphonate or primary alkyl sulphate of the formula above, or a mixture

thereof will be the desired non-soap anionic detergent and may provide 75 to 100wt% of the anionic non-soap detergent.

[0028] Examples of other non-soap anionic detergents which may be used include olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

The anionic detergent particles may contain some nonionic detergent. The anionic detergent particles may also contain minor ingredients such as water, sodium carboxymethylcellulose, fluorescers, dyes, etc.

[0029] Anionic detergent is preferably incorporated as particles which contain at least 20% of their own weight of anionic detergent, possibly a higher proportion such as at least 50% of their own weight. Particles which contain between 20 and 40% of anionic detergent can be prepared by spray-drying or granulation processes.

[0030] Processes for producing particles containing high concentrations of anionic detergent are set out in WO 96/06916A and WO 96/06917A (Unilever). In these processes, an aqueous paste containing an anionic detergent, or alternatively an acid detergent precursor and also an alkaline neutralising agent are fed into a drying zone where the paste material is heated to reduce the water content thereof, the dried material being subsequently cooled in a cooling zone to form detergent particles.

[0031] Desirably the drying zone is under a slight vacuum to facilitate the removal of water and volatiles. The vacuum may be from 100 Torr up to atmospheric pressure as this provides significant process flexibility. However, a vacuum in excess of 500 Torr up to atmospheric has the advantage of reducing capital investment whilst providing vacuum operation.

[0032] The process may be carried out in any suitable apparatus, but it is preferred that a flash reactor is employed. Suitable flash reactors include e.g. the Flash Drier system available from VRV Spa Impianti Industriali. The drying zone may have a heat transfer area of at least 10m². The cooling zone desirably has a heat transfer area of at least 5m².

[0033] As described in our WO97/32003A, the material in the cooling zone may be treated with a stream of cooling gas. Alternatively, finely divided non-detergent solid material, such as zeolite or silica particles, may be introduced into this zone to adhere to the surface of the particles. Such material may provide from 3 to 25% of the weight of the particles.

[0034] The above process routes can provide flash-dried detergent particles comprising at least 60% by weight of the particle of an anionic detergent and not more than 5% by weight of the particle of water.

[0035] These anionic detergent particles may comprise anionic detergent in an amount of at least 66% by weight of the particles, even better at least 70% but possibly not over 96%. The particles may have a porosity of from 0 to 25% by volume of the particle and a particle size distribution such that at least 80% of the particles have a particle size of 180-1500 microns. As mentioned the anionic detergent may be formed in situ by neutralisation of a free acid. The neutralising agent may be sodium hydroxide solution or sodium carbonate. However, in situ neutralisation is unlikely to be appropriate when the anionic detergent is primary alkyl sulphonate (PAS) because its acid form is unstable.

[0036] Anionic detergent particles may provide from 5% to at least 30% of the weight of the tablet or region of a tablet. The amount of them may be at least 8% or 10%. Their amount may be not over 20% of the weight of the tablet or region, especially when the particles contain at least 70 or 75% of their own weight of non-soap anionic detergent.

[0037] Other classes of organic detergent, such as amphoteric detergent, may be included but are not preferred. It is preferred that at least 50%, better at least 90% by weight of all non-soap organic detergent in the tablet or region of a tablet is contained either in the said particles which contain at least 40% nonionic detergent or in other particles which contain at least 20% of their own weight of non-soap organic detergent.

Disintegration Enhancing Particles

[0038] We have found and previously disclosed that the speed of disintegration of tablets can be enhanced by including certain materials. Thus our EP-A-839906 teaches that the speed of tablet disintegration can be enhanced by including sodium tripolyphosphate which is rich in the Phase I form of anhydrous sodium tripolyphosphate and may also be partially hydrated. Our EP-A-711827 and EP-A-838519 teach that the speed of disintegration of tablets with water-insoluble non-phosphorus builder can be accelerated by including highly water soluble materials, especially certain salts.

[0039] In preferred forms of this invention, a constituent of the tablet or region is particles containing material which serves to accelerate tablet disintegration in water and is either a material of high water-solubility or is a specified form of sodium tripolyphosphate, or a combination of the two. Such material may be present as at least 15 or 20% of the composition of a tablet or region thereof, possibly at least 25% up to 50, 55 or 60%, possibly more.

[0040] Highly water soluble materials, which are one of the two possibilities are compounds, especially salts, with a solubility at 20°C of at least 50 gms per 100 gms of water.

[0041] A solubility of at least 50 grams per 100 grams of water at 20°C is an exceptionally high solubility: many materials which are classified as water soluble are less soluble than this.

[0042] Some highly water-soluble materials which may be used are listed below, with their solubilities expressed as grams of solid to form a saturated solution in 100 grams of water at 20°C:-

Material	Water Solubility (g/100g)
Sodium citrate dihydrate	72
Potassium carbonate	112
Urea	>100
Sodium acetate, anhydrous	119
Sodium acetate trihydrate	76
Magnesium sulphate 7H ₂ O	71
Potassium acetate	>200

[0043] By contrast the solubilities of some other common materials at 20°C are:-

Material	Water Solubility (g/100g)
Sodium chloride	36
Sodium sulphate decahydrate	21.5
Sodium carbonate anhydrous	8.0
Sodium percarbonate anhydrous	12
Sodium perborate anhydrous	3.7
Sodium tripolyphosphate anhydrous	15

[0044] Preferably this highly water soluble material is incorporated as particles of the material in a substantially pure form (i.e. each such particle contains over 95% by weight of the material). However, the said particles may contain material of such solubility in a mixture with other material, provided that material of the specified solubility provides at least 50% by weight of these particles, better at least 80%.

[0045] Another possibility is that the said particles which promote disintegration are particles containing sodium tripolyphosphate with more than 50% of it (by weight of the particles) in the anhydrous phase I form. Such particles may contain at least 80% by weight tripolyphosphate and possibly at least 95%.

[0046] Sodium tripolyphosphate is very well known as a sequestering builder in detergent compositions. It exists in a hydrated form and two crystalline anhydrous forms. These are the normal crystalline anhydrous form, known as phase II which is the low temperature form, and phase I which is stable at high temperature. The conversion of phase II to phase I proceeds fairly rapidly on heating above the transition temperature, which is about 420°C, but the reverse reaction is slow. Consequently phase I sodium tripolyphosphate is metastable at ambient temperature.

[0047] A process for the manufacture of particles containing a high proportion of the phase I form of sodium tripolyphosphate by spray drying below 420°C is given in US-A-4536377.

[0048] Particles which contain this phase I form will often contain the phase I form of sodium tripolyphosphate as at least 55% by weight of the tripolyphosphate in the particles. Other forms of sodium tripolyphosphate will usually be present to a lesser extent. Other salts may be included in the particles, although that is not preferred.

[0049] Desirably, this sodium tripolyphosphate is partially hydrated. The extent of hydration should be at least 1% by weight of the sodium tripolyphosphate in the particles. It may lie in a range from 2.5 to 4%, or it may be higher, eg up to 8%.

[0050] Suitable material is commercially available. Suppliers include Rhone-Poulenc, France and Albright & Wilson, UK.

[0051] "Rhodiaphos HPA 3.5" from Rhone-Poulenc has been found particularly suitable. It is a characteristic of this grade of sodium tripolyphosphate that it hydrates very rapidly in a standard Olten test. We have found that it hydrates as quickly as anhydrous sodium tripolyphosphate, yet the prehydration appears to be beneficial in avoiding unwanted crystallisation of the hexahydrate when the material comes into contact with water at the time of use.

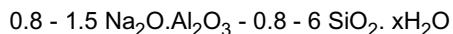
Detergency Builder

[0052] A tablet or tablet region will generally contain detergency builder. This may be sodium tripolyphosphate of the type just described. It may include sodium tripolyphosphate which has more of the phase form or is hydrated. It may be some other type of detergency builder. It may contain overall from 30 to 60% by weight sodium tripolyphosphate, reckoned as anhydrous.

[0053] Water-soluble phosphorous-containing inorganic detergency builders include the alkali-metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates, as well as sodium and potassium tripolyphosphates.

[0054] 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:



5 [0055] These materials contain some bound water (indicated as "xH₂O") and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO₂ units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

10 [0056] 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, and the novel maximum aluminium zeolite P described and claimed in EP 384070 (Unilever). This form of zeolite P is also referred to as zeolite MAP. One commercial form of it is denoted zeolite A24. Water insoluble detergency builder could be a layered sodium silicate as described in US 4664839.

15 [0057] NaSKS-6 is the trademark for a crystalline layered silicate marketed by Hoechst (commonly abbreviated as "SKS-6"). NaSKS-6 has the delta-Na₂SiO₅ 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, which can be used have the general formula NaMSi_xO_{2x+1}·yH₂O 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.

20 [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 and acrylic/maleic copolymers, monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono- di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates and hydroxyethyliminodiacetates.

25 [0059] Tablet compositions 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.

30 [0059] Builder materials may be incorporated as particles which contain from 40 to 80% by weight of builder, the balance being other material, probably not detergent. Such particles may provide 10 to 60% of the composition of the tablet or region of the tablet.

Proportions

35 [0060] Generally, a tablet made in accordance with this invention will contain overall from 2 or 5wt% up to 40 or 50wt% non-soap detergent, and from 5 or 10wt% up to 60 or 80wt% detergency builder. A discrete region of a heterogenous tablet may or may not contain these proportions of detergent and builder.

Other ingredients

40 [0061] Detergent tablets according to the invention may contain a bleach system. 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 tablet.

45 [0062] Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate. Bleach activators have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example tetraacetylene diamine (TAED), and perbenzoic acid precursors. The quaternary ammonium and phosphonium bleach activators disclosed in US 4751015 and US 4818426 (Lever Brothers Company) are also of interest. Another type of bleach activator which may be used, but which is not a bleach precursor, is a transition metal catalyst as disclosed in EP-A-458397, EP-A-458398 and EP-A-549272. A bleach system may also include a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

50 [0063] Bleach activator is usually present in an amount from 1 to 10% by weight of the tablet, possibly less in the case of a transition metal catalyst which may be used as 0.1 % or more by weight of the tablet.

55 [0064] The detergent tablets of the invention may also contain one of the detergency enzymes well known in the art for their ability to degrade various soils and stains and so aid in their removal. 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. Detergency enzymes are commonly employed in the form of particles or marumes, optionally with a protective coating, in amount of from about 0.1 % to about 3.0% by weight of the tablet.

[0065] The detergent tablets of the invention may also contain 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.

[0066] An antifoam material is advantageously included, especially if a detergent tablet is primarily intended for use in front-loading drum-type automatic washing machines. Antifoam materials in granular form are described in EP 266863A (Unilever). Such antifoam particles typically comprise a mixture of silicone oil, petroleum jelly, hydrophobic silica and alkyl phosphate as antifoam active material, sorbed onto a porous absorbed water-soluble carbonate-based inorganic carrier material.

[0067] 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 may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some detergency building. Preferably the detergent-rich particles contain from 5 to 15% silicate by weight of the particles. This improves the strength and free flow of these particles prior to tableting.

[0068] Further ingredients which can optionally be employed in fabric washing detergent tablet of the invention 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.

Tableting

[0069] Tableting entails compaction of a particulate composition which includes the detergent containing particles, the disintegration promoting particles and any other ingredients. A variety of tableting machinery is known, and can be used. Generally it will function by stamping a quantity of the particulate composition which is confined in a mould.

[0070] Tableting may be carried out without application of heat, so as to take place at ambient temperature or at a temperature above ambient. 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.

[0071] If any heat is supplied, it is envisaged that this will be supplied conventionally, such as by passing the particulate composition through an oven, rather than by any application of microwave energy.

Tablet size and density

[0072] 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, possibly 1100 gm/litre, up to 1450gm/litre or more. The tablet density may well lie in a range up to 1350 or 1400gm/litre.

[0073] The tablets may be made by a process comprising mixing said particles which themselves contain at least 45% of their own weight of nonionic detergent with other detergent ingredients, and compacting the resulting composition into tablets.

Example 1

[0074] Anionic detergent adjunct particles (LAS-A) containing 70% (of their own weight) of linear alkyl benzene sulphonate were prepared using a 1.2m² VRV Flash Drier, in the manner described in WO 97/32002. It had three equal jacket sections. Dosing ports for both liquids and powders were situated just prior to the first hot section, with mid-jacket dosing ports available in the final two sections. Zeolite was added via this port in the final section. An electrically-powered oil heater provided the heating to the first two jacket sections, with oil temperatures between 120°C and 190°C being used. Ambient process water at 15°C was used for cooling the jacket in the final section. Make-up air flow through the reactor was controlled between 10 and 50 m³/hr by opening a bypass on the exhaust vapour extraction fan. The motor was run at full speed, giving a top speed of about 30m/sec.

[0075] A mono pump was calibrated to dose ambient temperature LAS acid, and a peristaltic pump was calibrated to dose 47% sodium hydroxide. Screw feeders were calibrated to dose both sodium carbonate and zeolite A24. The sodium carbonate and the liquids were added just prior to the first hot section, but the zeolite was added into the third section which was cold.

[0076] The product was in the form of free-flowing particles containing

LAS-A: Ingredients	% by weight
Linear alkyl benzene sulphonate (LAS)	70%
Sodium carbonate	2%
Zeolite	25%
Non-detergent impurities and moisture	3%

5 [0077] Nonionic detergent particles (ND1) containing 56% of nonionic detergent were prepared by granulating nonionic detergent with silica and soap in an Eirich RV02 granulator. (For larger scale a Loedige recycler would be appropriate).

10 [0078] The silica was Sorbosil TC15 supplied by Crosfield, Warrington, UK. The nonionic detergent was warmed and mixed with fatty acid, then sprayed on to the silica in the granulator, while simultaneously spraying on sufficient alkali to neutralise the fatty acid. The product was cooled in a fluidised bed which also removed fines. Oversize particles (> 1400 μ m) were sieved out.

15 [0079] The resulting particles ND1 contained:

ND1: Ingredient	% by weight
Nonionic detergent	56%
Silica	30%
Soap and moisture	14%

20 [0080] Comparative nonionic detergent particles (ND2) were produced by granulating zeolite A24 which is maximum aluminium zeolite P from Crosfields with trisodium citrate in a Lödige recycler. Nonionic detergent was mixed with fatty acid and sprayed in while also spraying in sufficient 50% aqueous sodium hydroxide to neutralise the fatty acid. The resulting product contained

ND2 : Ingredient	% by weight
Zeolite A24	53.8
Sodium Citrate	7.9
Nonionic detergent	24.2
Soap	4.1
Water	10.0

25 [0081] Zeolite builder particles ZB1 were produced by continuously dosing zeolite A24, granular trisodium citrate and a 40% solution of acrylate/maleate copolymer (Sokolan CP5 solution) into a Lödige CB30 recycler. The CB30 was operated at 1500 rpm. The exiting powder was led through a Lödige KM300 ploughshare (120 rpm), in which densification took place. The resulting powder was dried in a fluid bed with an air temperature of 110°C. The composition of the resulting builder particle was:

ZB1 :Ingredient	% by weight
Zeolite A24	53.6
Trisodium Citrate	17.2
Sokolan CP5	19.0
Moisture	10.2

30 [0082] The detergent particles LAS-A and ND1 and the builder particles ZB1 and other ingredients were used to make tablets of formulation shown in the table below.

35 [0083] Comparative tablets used the ND2 particles in place of the ND1 particles.

		parts by weight	
		comparative	Example 1
5	Anionic detergent particles LAS-A	5.43	5.43
	Nonionic on zeolite particles ND2	6.91	-
	Nonionic on silica particles ND1	-	2.99
10	Zeolite particles ZB1	6.39	13.33
	Sodium carbonate	1.26	1.26
	Acrylate/maleate copolymer	0.53	0.53
15	Sodium disilicate	1.44	1.44
	Sodium carboxymethylcellulose particles	0.17	0.17
	Sodium percarbonate	5.92	5.92
20	TAED particles	2.09	2.09
	Sodium acetate trihydrate mixed with 1% of its own weight of zeolite	10.38	10.38
	Anti-foam particles	0.74	0.74
25	Sequestrant, fluorescer, soil-release polymer and coloured sodium carbonate particles	1.68	1.68
	TOTAL	42.94	45.96

[0084] Tablets were made containing 43 grams of the comparative composition, or 46 grams of the composition of Example 1. These tablets all contained 3.8 parts of alkylbenzene sulphonate, 1.67 parts of nonionic detergent, 8.5 parts of zeolite and 10.4 parts of sodium acetate trihydrate. The tablets were compacted with an applied force of 9.7 kN.

[0085] The speed of dissolution of the soluble constituents of the tablets was tested by means of a test procedure in which a tablet was placed on a plastic sieve with 2mm mesh size, immersed in 9 litres of tap water at ambient temperature of 20°C. The sieve was fastened to a stirrer, which rotated at 200 rpm. The water conductivity was monitored until it reached a constant value. The time for dissolution of the tablets was taken as the time (T₉₀) for change in the water conductivity to reach 90% of its final magnitude.

[0086] Tablet strength was tested by a procedure in which a cylindrical tablet is compressed radially between the platens of a materials testing machine until the tablet fractures. At failure, the tablet cracks and the applied force needed to maintain the displacement of the platens drops. Measurement is discontinued when the applied force needed to maintain the displacement has dropped by 25% from its maximum value.

[0087] The maximum force is the force at failure (F_f). From this measurement of force a test parameter called diametral fracture stress, was calculated using the equation

$$\sigma = 2 \frac{F_f}{\pi D t}$$

where

σ is the diametral fracture stress in Pascals,
F_f is the applied force in Newtons to cause fracture,
D is the tablet diameter in metres and
t is the tablet thickness in metres.

[0088] The force to cause fracture and the diametral fracture stress calculated from it are a direct assessment of strength and indicate the tablets' resistance to breakage when handled by a consumer at the time of use. The amount of energy (or mechanical work) put in prior to fracture is a measure of tablet deformability and is relevant to the tablets' resistance to breakage during transport. This energy or work prior to failure is assessed as the "break energy" which is

the area under a graph of force against displacement, up to the point of break. It is given by the equation:

$$5 \quad E_b = \int_0^{x_f} F(x) dx$$

where

10 E_b is the break energy in joules,
 x is the displacement in metres,
 F is the applied force in Newtons at displacement x, and
 x_f is the displacement at failure.

15 [0089] The values of dissolution time, fracture stress and break energy are set out in the following table:

	Comparative	Example 1
DFS (kPa)	18.9	21.5
T_{90} (sec)	123	120
E_b (mJ)	9.2	17.3

25 [0090] It can be seen that break energy of Example 1 tablets was better than that of the comparative tablets while other properties were similar.

Examples 2 and 3

30 [0091] Anionic detergent adjunct particles (LAS-B) were made as described in Example 1, but with a higher content of detergent. They contained

LAS-B: Ingredient	% by weight
Linear alkyl benzene sulphonate	82%
Sodium carbonate	4%
Zeolite	10%
Non-detergent impurities and moisture	4%

40 [0092] The above particles LAS-B and the nonionic particles ND1 described in the previous example were mixed with other materials to make two detergent compositions set out in the table below. These included particles of sodium tripolyphosphate specified to contain 70% phase I form and contain 3.5% water of hydration (Rhodia-Phos HPA 3.5 available from Rhone-Poulenc).

	Parts by Weight	
	Example 2	Example 3
Anionic detergent particles (LAS-B)	13.5	13.5
Nonionic detergent particles (ND1)	8.9	8.9
Rhodiaphos HPA3.5 tripolyphosphate	46.65	30.2
Acrylate/maleate copolymer	1.5	1.5
Sodium silicate	4.0	4.0
Sodium carboxymethylcellulose particles (SCMC)	0.3	0.3
Fluorescer on inert carrier	0.15	0.15

Table continued

		Parts by Weight	
		Example 2	Example 3
5	Sodium percarbonate	15.1	15.1
10	TAED particles	3.4	3.4
15	Anti-foam particles	3.2	3.2
20	Sequestrant, soil-release polymer and coloured sodium carbonate particles	2.7	2.7
25	TOTAL	99.4	82.95

[0093] A comparative detergent composition was made, starting with a spray-dried base powder (BP1) of the following composition:

BP1 : Ingredient	Parts by Weight
Sodium linear alkylbenzene sulphonate	11.0
C ₁₃₋₁₅ fatty alcohol 7EO	2.6
C ₁₃₋₁₅ fatty alcohol 3EO	2.4
Soap	0.2
Sodium tripolyphosphate*	16.9
Acrylate/maleate copolymer	1.5
Sodium silicate	4.0
Sodium carboxymethylcellulose particles	0.3
Fluorescer on inert carrier	0.15
moisture and impurities	5.95
TOTAL	45
Added to the slurry as anhydrous sodium tripolyphosphate containing at least 70% phase II form.	

[0094] This powder was mixed with other ingredients as follows:

Ingredient	Parts by Weight
Base powder (BP1)	45
Rhodiaphos HPA3.5 tripolyphosphate	30.2
Sodium percarbonate	15.1
TAED particles	3.4
Anti-foam particles	3.2
Sequestrant, soil-release polymer and coloured sodium carbonate particles	2.7
TOTAL	99.6

[0095] These compositions are also set out alongside each other in the following table

			Parts by weight		
Ingredient			Example 2	Example 3	Comparative
5	Sodium linear alkylbenzene sulphonate		11.0	11.0	11.0
Nonionic detergent			5.0	5.0	5.0
10	aluminosilicate		1.35	1.35	0
silica			2.65	2.65	0
15	Rhodiaphos HPA3.5 tripolyphosphate		46.65	30.2	30.2
other tripolyphosphate			0	0	16.9
20	Acrylate/maleate copolymer		1.5	1.5	1.5
Sodium silicate			4.0	4.0	4.0
25	Sodium percarbonate		15.1	15.1	15.1
TAED particles			3.4	3.4	3.4
Anti-foam particles			3.2	3.2	3.2
SCMC and fluorescer on carrier			0.45	0.45	0.45
30	Sequestrant, soil-release polymer and coloured sodium carbonate particles		2.7	2.7	2.7
Soap, sodium carbonate, moisture and other impurities			2.35	2.35	6.15
25	Total		99.35	82.9	99.6

[0096] 40 gram portions of each composition were stamped into cylindrical tablets of 44 mm diameter. Various amounts of compaction force were used. The composition of Example 2 was also stamped into 32 gram tablets, so as to provide tablets of this composition containing the same amount of detergent as the 40 gram comparative tablets.

[0097] The tablets were tested as in Example 1. The results are shown in the tables below, arranged to show comparison of tablets with similar diametral fracture stress (DFS):

Compaction force (kN)	Example 2 as 40 gram tablets			Comparative as 40 gram tablets		
	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)
30	55.8	180	20.5			
9.7				54.2	400	15
40	15	27.4	13.9			
9.7	18.6	105	6.7			
5.0				20.9	220	6.4
45	5.0	7.4	2.8			
2.5				6.5	190	2.25

Compaction force (kN)	Example 3 as 40 gram tablets			Example 3 as 32 gram tablets			Comparative as 40 gram tablets		
	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)
50	9.7						54.2	400	15
55	15	39.1	250	20.3	36.1	275	22.9		
7.5							36.8	310	10.5
9.7	29.9	200	15.2	23.4	240	14.1			

Table continued

5	Compaction force (kN)	Example 3 as 40 gram tablets			Example 3 as 32 gram tablets			Comparative as 40 gram tablets		
		DFS (kPa)	T ₉₀ (sec)	E _b (mJ)	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)
5.0								20.9	220	6.4
5.0	10.8	145	6.1	9.8	140	5.7				
2.5								6.5	190	2.25

[0098] It is apparent that the invention makes it possible to increase break energy, reduce dissolution time and/or reduce the tablet weight which is needed to deliver the same quantity of detergent.

15 Example 4

[0099] Adjunct particles LAS-B and ND1 as described in the preceding examples were used to make tablets of the following formulation:

		Parts by Weight	
20	Anionic detergent particles (LAS-B)		16.0
25	Nonionic detergent particles (ND1)		10.0
30	Rhodiaphos HPA3.5 tripolyphosphate		48.0
35	Acrylate/maleate copolymer		2.0
	Sodium silicate		4.0
	Sodium carboxymethylcellulose particles		0.5
	Polyvinylpyrrolidone		1.0
	Sodium carbonate		7.0
	Sodium sulphate		5.0
	Anti-foam particles		3.5
	Sequestrant, soil-release polymer and coloured sodium carbonate particles		3.0
	TOTAL		100

40 Example 5

[0100] Adjunct particles (LAS-B and ND1) as described in previous Examples were used together with further ingredients to make 40 gram tablets with two layers of unequal weight (10 grams and 30 grams). The overall formulation was similar to Example 2 but contained slightly more alkylbenzene sulphonate and slightly less tripolyphosphate. A base powder (BP1A) with the same composition as used in Examples 2 and 3 but taken from a different batch, was used to make comparative tablets with two layers of unequal weight. The overall formulation was the same as for the previous comparative tablets.

[0101] When making these two layer tablets, the composition for one layer was placed in a mould and lightly compacted, the composition for the other layer was then added to the mould, and compaction force was applied to the mould contents.

50 [0102] The formulations are set out in the table below:

55		Parts by Weight					
		Comparative			Example 5		
		thin layer	thick layer	total	thin layer	thick layer	total
	Base powder (BP1A)	4.57	13.6	18.17	-	-	-

Table continued

	thin layer	thick layer	total	thin layer	thick layer	total	
5	Anionic detergent particles (LAS-B)	-	-	-	1.59	4.73	6.32
10	Nonionic detergent particles (ND1)	-	-	-	0.90	2.67	3.57
15	Rhodiaphos HPA3.5 tripolyphosphate	3.05	9.07	12.12	4.51	13.42	17.93
20	Acrylate/maleate copolymer	-	-	-	0.15	0.45	0.6
25	Sodium silicate	-	-	-	0.40	1.20	1.6
30	SCMC particles, fluorescer and soil release polymer	-	-	-	0.07	0.20	0.27
35	Sodium percarbonate	0	6.05	6.05	0	6.05	6.05
40	TAED particles	1.36	0	1.36	1.36	0	1.36
45	Anti-foam particles	0	1.28	1.28	0	1.28	1.28
50	Sequestrant, and coloured sodium carbonate particles	1.02	0	1.02	1.02	0	1.02
55	TOTAL	10	30	40	10	30	40

[0103] The tablets and comparative tablets were tested in the same ways as for Example 1 above, with the following results:

Compaction force (kN)	Example 5			Comparative two layer tablets		
	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)	DFS (kPa)	T ₉₀ (sec)	E _b (mJ)
30	55.8	120	41.7			
20	43.1	115	28.0			
9.7	21.4	105	10.6	52.1	275	15.7
5.0	8.2	105	2.7	21.8	210	7.1
2.5				7.0	135	2.6

Claims

1. A detergent tablet of compressed particulate composition, wherein the tablet or a region thereof comprises organic detergent and water-soluble detergency builder, **characterised in that** the tablet or region thereof is compacted from a composition which contains:
 - (i) particles which themselves contain at least 45% of their own weight of nonionic detergent, the particles contain an inorganic water-insoluble carrier material, and the nonionic is primary or secondary alcohol ethoxylate, and
 - (ii) other ingredients.
2. A tablet according to claim 1 wherein the composition contains particles which contain at least 50% by weight of nonionic detergent.
3. A tablet according to claim 1 or claim 2 wherein the composition of said tablet or region contains from 3 is to 40% by weight of nonionic detergent, at least half of which is present as said particles.
4. A tablet according to any one of the preceding claims wherein the composition of said tablet or region also includes particles containing at least 20% of their own weight of non-soap anionic detergent.

5. A tablet according to claim 4 wherein the composition of said tablet or region contains from 5 to 50% by weight of non-soap detergent, at least half of which is present as said particles containing at least 45% by weight of nonionic detergent or as particles containing at least 20% of their own weight of other non-soap detergent.
- 5 6. A tablet according to any one of the preceding claims wherein at least 90% of the non-soap organic detergent in the tablet or region thereof is contained in said particles which contain at least 45% of their own weight of nonionic detergent or in particles which contain at least 20% of their own weight of other non-soap detergent.
- 10 7. A tablet according to any one of the preceding claims wherein the composition of said tablet or region includes particles containing at least 80% of their own weight of one or more water-soluble materials selected from compounds with a water-solubility exceeding 50 grams per 100 grams water at 20°C, sodium tripolyphosphate containing at least 50% of its own weight of the phase I anhydrous form, and mixtures thereof.
- 15 8. A tablet according to claim 7 the composition of said tablet or region contains from 15 to 60% by weight of said particles containing at least 80% of their own weight of one or more water-soluble materials.
9. A tablet according to claim 7 the composition of said tablet or region contains from 25 to 50% by weight of said particles containing at least 80% of their own weight of one or more water-soluble materials.
- 20 10. A tablet according to any one of claims 7 to 9 wherein the composition of the tablet or region contains from 20 to 60% by weight of said tripolyphosphate which is partially hydrated so as to contain water of hydration in an amount which is at least 1% by weight of the sodium tripolyphosphate.
- 25 11. A tablet according to any one of claims 7 to 9 wherein the composition of the tablet or region contains to 55% by weight of particles which contain at least 95% of their own weight of said tripolyphosphate, which is partially hydrated so as to contain water of hydration in an amount which is at least 1% by weight of the sodium tripolyphosphate.
- 30 12. A tablet according to any one of claims 7 to 9 wherein the composition of the tablet or region contains at least 20% by weight of said particles which themselves contain at least 80% of their own weight of one or more salts with a water-solubility exceeding 50 grams per 100 grams water at 20°C.
13. A tablet according to claim 12 wherein said compound is one of more of partially or completely hydrated sodium citrate, partially or completely hydrated sodium acetate, and potassium acetate.
- 35 14. A tablet according to any one of the preceding claims wherein, overall, said tablet contains from 5 to 40% of non-soap organic detergent and from 15 to 80% of detergency builder.
15. A tablet according to claim 14 containing overall from 30 to 60% by weight sodium tripolyphosphate, reckoned as anhydrous.
- 40 16. A tablet according to any one of the preceding claims wherein the composition of the tablet or region thereof contains from 10 to 60% of particles which contain from 40 to 80% of their own weight of detergency builder and 20 to 60% of their own weight of other, non-detergent, material.
- 45 17. A process for making a tablet as defined in any one of the preceding claims which process comprises mixing said particles which themselves contain at least 45% of their own weight of nonionic detergent with other detergent ingredients, and then compacting the resulting composition into tablets.

50 Patentansprüche

1. Waschmitteltablette aus verdichteter, teilchenförmiger Zusammensetzung, wobei die Tablette oder ein Bereich davon organisches Waschmittel und in Wasser löslichen Waschmittelbuilder umfasst, **dadurch gekennzeichnet, dass** die Tablette oder der Bereich davon aus einer Zusammensetzung verdichtet ist, die enthält:
 - 55 (i) Teilchen, die selbst mindestens 45% ihres Eigengewichts nichtionisches Waschmittel enthalten, wobei die Teilchen ein anorganisches, in Wasser unlösliches Trägermaterial enthalten, und das Nichtionische primäres oder sekundäres Alkoholethoxylat ist, und

(ii) weitere Bestandteile.

2. Tablette nach Anspruch 1, wobei die Zusammensetzung Teilchen enthält, die mindestens 50 Gew.-% nichtionisches Waschmittel enthalten.
- 5 3. Tablette nach Anspruch 1 oder 2, wobei die Zusammensetzung der Tablette oder des Bereichs 3 bis 40 Gew.-% nichtionisches Waschmittel enthält, wobei mindestens die Hälfte davon als die Teilchen vorliegt.
- 10 4. Tablette nach einem der vorangehenden Ansprüche, wobei die Zusammensetzung der Tablette oder des Bereichs auch Teilchen einschließt, die mindestens 20% ihres Eigengewichts anionisches Nicht-Seifen-Waschmittel enthalten.
- 15 5. Tablette nach Anspruch 4, wobei die Zusammensetzung der Tablette oder des Bereichs 5 bis 50 Gew.-% Nicht-Seifen-Waschmittel enthält, wobei mindestens die Hälfte davon als die Teilchen vorliegt, die mindestens 45 Gew.-% des nichtionischen Waschmittels enthalten oder als Teilchen, die mindestens 20% ihres Eigengewichts anderes Nicht-Seifen-Waschmittel enthalten.
- 20 6. Tablette nach einem der vorangehenden Ansprüche, wobei mindestens 90% des organischen Nicht-Seifen-Waschmittels in der Tablette oder dem Bereich davon in den Teilchen, die mindestens 45% ihres Eigengewichts nichtionisches Waschmittel enthalten oder in Teilchen, die mindestens 20% ihres Eigengewichts anderes Nicht-Seifen-Waschmittel enthalten, enthalten sind.
- 25 7. Tablette nach einem der vorangehenden Ansprüche, wobei die Zusammensetzung der Tablette oder des Bereichs Teilchen einschließt, die mindestens 80% ihres Eigengewichts von einem oder mehreren in wasserlöslichen Materialien, ausgewählt aus Verbindungen mit einer Wasserlöslichkeit, die 50 g pro 100 g Wasser bei 20°C übersteigt, Natriumtripolyphosphat, das mindestens 50% seines Eigengewichts der wasserfreien Phase I-Form enthält und Gemischen davon, enthalten.
- 30 8. Tablette nach Anspruch 7, wobei die Zusammensetzung der Tablette oder des Bereichs 15 bis 60 Gew.-% der Teilchen enthält, die mindestens 80% ihres Eigengewichts von einem oder mehreren in Wasser löslichen Materialien enthalten.
- 35 9. Tablette nach Anspruch 7, wobei die Zusammensetzung der Tablette oder des Bereichs 25 bis 50 Gew.-% der Teilchen enthält, die mindestens 80% ihres Eigengewichts von einem oder mehreren in Wasser löslichen Materialien enthalten.
- 40 10. Tablette nach einem der Ansprüche 7 bis 9, wobei die Zusammensetzung der Tablette oder des Bereichs 20 bis 60 Gew.-% des Tripolyphosphats enthält, welches teilweise hydratisiert ist, sodass Hydratationswasser in einer Menge enthalten ist, die mindestens 1 Gew.-% des Natriumtripolyphosphats beträgt.
- 45 11. Tablette nach einem der Ansprüche 7 bis 9, wobei die Zusammensetzung der Tablette oder des Bereichs bis 55 Gew.-% der Teilchen enthält, die mindestens 95% ihres Eigengewichts von dem Tripolyphosphat enthalten, welches teilweise hydratisiert ist, sodass Hydratationswasser in einer Menge enthalten ist, die mindestens 1 Gew.-% des Natriumtripolyphosphats beträgt.
- 50 12. Tablette nach einem der Ansprüche 7 bis 9, wobei die Zusammensetzung der Tablette oder des Bereichs mindestens 20 Gew.-% der Teilchen enthält, die selbst mindestens 80% ihres Eigengewichts von einem oder mehreren Salzen mit einer Wasserlöslichkeit, die 50 g/100 g Wasser bei 20°C übersteigt, enthalten.
- 55 13. Tablette nach Anspruch 12, wobei die Verbindung eine oder mehrere von teilweise oder vollständig hydratisiertem Natriumcitrat, teilweise oder vollständig hydratisiertem Natriumacetat und Kaliumacetat ist.
14. Tablette nach einem der vorangehenden Ansprüche, wobei die Tablette insgesamt 5 bis 40% organisches Nicht-Seifen-Waschmittel und 15 bis 80% Waschmittelbuilder enthält.
15. Tablette nach Anspruch 14, die insgesamt 30 bis 60 Gew.-% Natriumtripolyphosphat, als wasserfrei berechnet, enthält.

16. Tablette nach einem der vorangehenden Ansprüche, wobei die Zusammensetzung der Tablette oder des Bereichs davon 10 bis 60% Teilchen enthält, die 40 bis 80% ihres Eigengewichts Waschmittelbuilder und 20 bis 60% ihres Eigengewichts von anderem Nicht-Waschmittelmateriel enthält.

5 17. Verfahren zur Herstellung einer Tablette, wie in einem der vorangehenden Ansprüche definiert, wobei das Verfahren Vermischen der Teilchen, die selbst mindestens 45% ihres Eigengewichts nichtionisches Waschmittel enthalten, mit anderen Waschmittelbestandteilen, und anschließend Verdichten der erhaltenen Zusammensetzung zu Tabletten umfasst.

10

Revendications

1. Comprimé détergent de composition particulaire comprimée, dans lequel le comprimé ou une région de celui-ci comprend du détergent organique et de l'édificateur de détergence soluble dans l'eau, **caractérisé en ce que** le comprimé ou une région de celui-ci est compacté à partir d'une composition qui contient :

15 (i) des particules qui elles mêmes contiennent du détergent non ionique à hauteur d'au moins 45 % de leur propre poids, les particules contenant un matériau formant support inorganique non soluble dans l'eau, et le composant non ionique étant de l'éthoxylat d'alcool primaire ou secondaire ; et

20 (ii) d'autres ingrédients.

2. Comprimé selon la revendication 1, dans lequel la composition contient des particules qui contiennent au moins 50 % en poids de détergent non ionique.

25 3. Comprimé selon la revendication 1 ou la revendication 2, dans lequel la composition dudit comprimé ou de ladite région contient de 3 à 40 % en poids de détergent non ionique, au moins la moitié étant présent sous la forme desdites particules.

30 4. Comprimé selon l'une quelconque des revendications précédentes, dans lequel la composition dudit comprimé ou de ladite région inclut également des particules contenant du détergent anionique non savon au moins à hauteur de 20 % de leur propre poids.

35 5. Comprimé selon la revendication 4, dans lequel la composition dudit comprimé ou de ladite région contient 5 à 50 % en poids de détergent non savon, dont au moins la moitié est présent sous la forme desdites particules contenant au moins 45 % en poids de détergent non ionique ou sous la forme de particules contenant au autre détergent non savon au moins à hauteur de 20 % de leur propre poids.

40 6. Comprimé selon l'une quelconque des revendications précédentes, dans lequel au moins 90 % du détergent organique non savon dans le comprimé ou dans une région de celui-ci est contenu dans lesdites particules qui contiennent du détergent non ionique à hauteur d'au moins 45 % de leur propre poids ou dans des particules qui contiennent un autre détergent non savon à hauteur d'au moins 20 % de leur propre poids.

45 7. Comprimé selon l'une quelconque des revendications précédentes, dans lequel la composition dudit comprimé ou de ladite région inclut des particules contenant au moins 80 % de leur propre poids d'un ou de plusieurs matériaux solubles dans l'eau sélectionnés à partir de composés ayant une solubilité dans l'eau dépassant 50 grammes pour 100 grammes d'eau à 20°C, du tripolyphosphate de sodium contenant de la forme en phase 1 anhydre à hauteur d'au moins 50 % de son propre poids, et des mélanges de ceux-ci.

50 8. Comprimé selon la revendication 7, dans lequel la composition dudit comprimé ou de ladite région contient de 15 à 60 % en poids desdites particules contenant un ou plusieurs matériaux solubles dans l'eau à hauteur d'au moins 80 % de leur propre poids.

55 9. Comprimé selon la revendication 7, dans lequel la composition dudit comprimé ou de ladite région contient de 25 à 50 % en poids desdites particules contenant un ou plusieurs matériaux solubles dans l'eau à hauteur d'au moins 80 % de leur propre poids.

10. Comprimé selon l'une quelconque des revendications 7 à 9, dans lequel la composition du comprimé ou de la région contient de 20 à 60 % en poids dudit tripolyphosphate qui est partiellement hydraté de façon à contenir de l'eau

d'hydratation dans une quantité qui est d'au moins 1 % en poids du tripolyphosphate de sodium.

- 5 11. Comprimé selon l'une quelconque des revendications 7 à 9 dans lequel la composition du comprimé ou de la région contient jusqu'à 55 % en poids de particules qui contiennent ledit tripolyphosphate à hauteur d'au moins 95 % de leur propre poids, ledit tripolyphosphate étant partiellement hydraté pour contenir de l'eau d'hydratation dans une quantité qui est d'au moins 1 % en poids du tripolyphosphate de sodium.
- 10 12. Comprimé selon l'une quelconque des revendications 7 à 9, dans lequel la composition du comprimé ou de la région contient au moins 20 % en poids desdites particules qui elles-mêmes contiennent un ou plusieurs sels à hauteur d'au moins 80 % de leur propre poids, ledit ou lesdits sels ayant une solubilité dans l'eau supérieure à 50 grammes pour 100 grammes d'eau à 20°C.
- 15 13. Comprimé selon la revendication 12 dans lequel ledit composé est un composé sélectionné parmi le citrate de sodium partiellement ou entièrement hydraté, l'acétate de sodium partiellement ou entièrement hydraté et l'acétate de potassium.
- 20 14. Comprimé selon l'une quelconque des revendications précédentes, dans lequel, dans l'ensemble, ledit comprimé contient de 5 à 40 % d'un détergent organique non savon et de 15 à 80 % d'édificateur de détergence.
- 25 15. Comprimé selon la revendication 14 contenant dans l'ensemble de 30 à 60 % en poids de tripolyphosphate de sodium reconnu pour être anhydre.
16. Comprimé selon l'une quelconque des revendications précédentes, dans lequel la composition du comprimé ou d'une région de celui-ci contient de 10 à 60 % de particules qui contiennent de l'édificateur de détergence à hauteur de 40 à 80 % de leur propre poids et un autre matériau non détergent à hauteur de 20 à 60 % de leur propre poids.
- 30 17. Procédé de fabrication d'un comprimé tel que défini dans l'une quelconque des revendications précédentes, ledit procédé comprenant les étapes consistant à mélanger lesdites particules qui elles-mêmes contiennent du détergent non ionique à hauteur d'au moins 45 % de leur propre poids avec d'autres ingrédients détergents, puis à compacter en comprimés la composition qui en résulte.

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