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(71) Applicants:
- **UNILEVER N.V.**
  3013 AL Rotterdam (NL)
  Designated Contracting States:
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- **UNILEVER PLC**
  London EC4P 4BQ (GB)
  Designated Contracting States:
  IE GB

(72) Inventors:
- **Boskamp, Jelles Vincent**
  Unilever R&D Vlaardingen
  3133 AT Vlaardingen (NL)
- **Callaghan, Ian Charles**
  Unilever R&D Vlaardingen
  3133 AT Vlaardingen (NL)

(74) Representative:
Rosen Jacobson, Frans Lucas M. et al
Unilever N.V.,
Patent Division,
P.O. Box 137
3130 AC Vlaardingen (NL)

(54) Cleaning compositions

(57) The invention provides disintegrant granules, comprising STS and/or SCS, a water insoluble inorganic material, a water swelling clay and a water swellable agent, the latter in amounts of 1 to 10 wt % on total weight of the granule, further embodiments of the invention provide tablets wherein the above granules are present.

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Description

[0001] The present invention relates to novel tablets comprising disintegrant granules and to methods for making these. These tablets are intended to disintegrate when placed in water and thus are intended to be consumed in a single use. The tablets may be suitable for use in machine dishwashing, the washing of fabrics or other cleaning tasks.

[0002] Products in tablet form have several advantages over powdered products: for example, they do not require measuring and are thus easier to handle and to dispense into the wash-load, and they are more compact, hence facilitating more economical storage.

[0003] Detergent compositions in tablet form and intended for fabric washing have been described in a number of patent documents including, for example GB 911204, EP-A-711827, WO-98/42817 and WO-99/20730 (all Unilever), GB 1,029,263 (Procter & Gamble), US 3953350 (Kao), DE 19 637 606 (Henkel). Tablets of composition suitable for machine dishwashing have been disclosed in EP-A-318204, WO96/23530 and US-A-5691293.

[0004] Tablets of a cleaning composition are generally made by compressing or compacting a composition in particulate form. Although it is desirable that tablets have adequate strength when dry, they should disperse and dissolve quickly when brought into contact with water and it can be difficult to obtain both properties together. Tablets formed using a low compaction pressure tend to crumble and disintegrate on handling and packing in particular after prolonged storage periods; while more forcefully compacted tablets may be sufficiently cohesive but then fail to disintegrate or disperse to an adequate extent in the wash. Tabletting will often be carried out with enough pressure to achieve a compromise between these desirable but antagonistic properties. However, it remains desirable to improve in particular the storage properties of the tablets.

[0005] US-A-3018267 (Procter & Gamble) taught that the force, and hence pressure, applied when compacting a composition into tablets should be limited, or else the tablets would take too long to dissolve.

[0006] If a tablet contains organic surfactant, this can function as a binder, plasticising the tablet. However, it can also 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 both good strength and speed of disintegration: the problem has proved especially acute with tablets formed by compressing powders containing surfactant and built with insoluble detergency builder such as sodium aluminosilicate (zeolite).

[0007] It is known to include highly soluble materials whose function is to enhance disintegration of tablets when placed in wash water. Some tablets which are sold commercially incorporate urea for this purpose. EP-A-711 827 (Unilever) teaches the use of sodium citrate for this purpose and EP-A-838519 and WO 00/32741 (both Unilever) teach the use of sodium acetate trihydrate for this purpose.

[0008] EP 971028 and EP 971029 (both P&G patent) disclose the use of a hydrotrope that enhances strength with at least 30 %. Although STS is indicated as an example hereof we found that when applying STS for this purpose this could not enhance this strength with more than 30 % and thus is STS not part of the disclosure for the use in the granules nor in the base powder of tablets.

[0009] A number of documents have taught that the disintegration of tablets of cleaning composition can be accelerated by incorporating in the tablet a quantity of a water-insoluble but water-swellable material serving to promote disintegration of the tablet when placed in water at the time of use. Such documents include WO98/40462 (Rettenmaier), WO98/55583 (Unilever) and WO-98/40463 (Henkel). DE 199 32 569 (Henkel) discloses the use of auxiliary disintegrant agents of a fine particle size in machine dishwashing tablets.

[0010] Typical water-swellable agents which have been disclosed as possible tablet disintegrating agents are starches, cellulose and cellulose derivatives, alginates, dextrans, cross-linked polyvinyl pyrrolidones, gelatines and formaldehyde casein as well as a wide variety of clay minerals and certain ionexchange resins.

[0011] Often these water swellable agents have no function in fabric washing except to aid tablet disintegration. Furthermore, because they are insoluble and of relatively large particle size, they tend to deposit on fabric during the wash; see for example WO98/55575 (Henkel). As a result, several attempts have been made to minimise the deposition of these disintegrants, for example by combining such a water swellable water insoluble disintegrant with a second, highly soluble disintegration aid (see WO98/55582 and WO 98/55590 both Unilever). Other attempts have included the use of a preferred particle size of the disintegrant. For example, WO98/55583 (Unilever) discloses the use of such materials at a particle dimension of at least 400um to give more efficient disintegration. WO98/55575 and DE 199 01 063 (both Henkel) however teach the use of cellulose disintegrating aids with a particle size of less than 100 um in order to minimise deposition. For DE 199 01 063, the cellulose material is mixed with a material of a given oil absorbing capacity, which includes some zeolites.

[0012] According to EP 1 035 198 perfumed tablets that comprise a clay have a problem with its performance due to the presence of clay.These problems are said to be overcome by the use of a coating wherein the clay is incorporated while the perfume is provided with a Schiff's base. Although the tablet can contain a component with a cohesive effect such as sodium di-isoalkylbenzenesulphonates this component is not examplified in the examples ,herein the use of 0.5 wt % of STS in the Blown Powder (examples Q,T and U) is indicated but it remains unclear what the function of

this STS is.Our tablets are free of a coating and do not contain a Schiff's base in the perfume composition.A similar teaching can be derived from EP 1 035 199 although it is stated herein that the use of a sequestrant in the coating helps to overcome the problems.

[0013] In DE 198 18 965 tablets are disclosed that comprise detergents and builders and wherein an aromatic sulphonate is incorporated.This sulphonate can also be STS or SCS.These tablets are however free of clay and therefore do not provide a solution for tablets that contain clay.Further it can be concluded from this DE that the sulphonate is only present in the granules (the examples illustrate that the best balance between hardness and dispersabilty is obtained when the sulphonate is present in the granules).We found that the best balance between strength / dispersabilty and the avoidance of residues on the washed products is obtained when a clay is used in the tablets in combination with the use of STS and / or SCS in the matrix material.

DE 199 50 765 teaches tablets containing 60-95 wt% of anionic surfactants ; 5-40 wt% of a hydrotrope and 0-35 wt% of a carrier.These tablets have a better stability (are less friable during storage).Hydrotropes that can be applied are sulphonates including STS and SCS.These components function as a solubility aid i.e they help making less soluble components better soluble in water.These sulphonates are used in amounts of 6-35 wt%.The presence of clay is not mentioned in this document and therefore this patent does not provide a solution for tablets wherein clay is present.

[0014] Thus, there is a need to provide a cleaning composition in the form of a tablet, which has adequate strength when dry so it withstands handling during manufacture, packaging, transport and storage, yet which disperses and dissolves in an acceptable time when brought into contact with a washing medium such as water, but, without causing the additional problem of unacceptable residues on the substrate being cleaned.

The process and the tablets

[0015] Surprisingly, we have now found that if a particular agent is incorporated into a tablet, either as part of a disintegrating granule or as part of the matrix material or both and the tablet preferably also comprises certain water-soluble disintegration-promoting particles,in addition to non-soap surfactants , and detergency builder, then the storage properties of the tablets are improved considerably. The tablets are made by a specific process , which process comprises mixing disintegrant granules comprising a water-swelling clay, a water-insoluble inorganic material and a water swellable agent and optionally the STS and/or SCS with the other constituents of the detergent composition, optionally comprising the STS and/or SCS to produce a particulate detergent composition comprising STS and/or SCS, placing a quantity of the resultant particulate detergent composition within a mould, and compacting the composition within the mould to produce the tablet.

This process will result in tablets of compacted particulate detergent composition comprising non-soap surfactant and detergency builder, wherein the tablet or a discrete region thereof comprises a matrix material and disintegrant granules comprising a water-insoluble inorganic material and a water-swellable agent and optionally a water-swelling clay, while either the granules or the matrix material or both comprise an agent, selected from the group consisting of STS and SCS

[0016] According to a preferred embodiment we perform our process, while the disintegrant granules also comprise a water-swellable agent which, in its anhydrous state, comprises no more than 20 per cent , preferably 1 to 20 wt %, more preferably 1 to 10 wt % of the combined weight of, the water insoluble material , the STS and / or SCS and the water-swellable agent and if present the swelling clay.

A suitable method for carrying out the dry granulation process is a process comprising the blending of the ingredients of the granular composition in a mixer followed by roller compaction of the mixture so produced. During the roller compaction suitable roller pressures of 2 to 25 Mpa are applied. The screening of the granules after the roller compaction is performed to collect particles with size of 500 to 3000 microns , preferably 700 to 1200 microns (measured by sieve analysis).

[0017] The STS and / or SCS is applied in amounts of 0.5 to 22 wt % on total tablet. If applied in the matrix material the amount of STS and / or SCS ranges from 0.5 to 5 wt % on total tablet and when applied in the granules this amount is 0.15 to 50 wt %.

[0018] The swelling clay, if applied in our tablets, preferably is a bentonite clay. The water insoluble inorganic material is suitably silica, a material containing at least 70 weight per cent silica or a zeolite. The water content of the zeolite can be as high as 22 wt%. Preferred zeolites are zeolite P having a water content in the range of 9 to 12 wt% of the zeolite. In the instance that water swelling clay and zeolite are both applied in the granules it is preferred to use them in a ratio clay : zeolite of 9 : 1 to 1 : 9 by weight. The amounts of water swelling clay repectively water insoluble inorganic material in the granules are respectively 0 to less than 50 wt % and 35 to 70 wt%. Preferred water swellable agents that are applied have a water-swelling capacity of at least 5 cm^3/g and are selected from the group consisting of cellulose, cross-linked cellulose, carboxymethyl cellulose, sodium carboxymethyl cellulose, cross-linked sodium carboxymethyl cellulose, pre-gelatinised starch, cross linked starch, or cross linked polyvinyl pyrrolidone Currently preferred are Aquasorb A500 (ex Hercules) and Ac-Di-Sol and Nilyn XL90 (ex FMC Corporation, USA). These water swellable agents are preferably present in the granules in amounts of less than 2 wt% (on total tablet).

[0019] The tablets according to the present invention 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 the particles of that composition will 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 heterogeneous tablet according to the present invention, each discrete region of the tablet will preferably have a mass of at least 5 grams.

[0020] Unless otherwise stated, all references to percentages herein are to percentages by weight based upon the total weight of the tablet, or region thereof.

[0021] The water-swellable agent which is present comprises in its anhydrous state 1 to 20% by weight of the combined weight of said water insoluble inorganic material, said STS and/or SCS, said water-swellable agent and said water swelling clay, if present.

[0022] By "water-insoluble" as used herein, in relation to the inorganic material, is meant a compound with a solubility in water at 25°C of less than 5 grams per 100 grams of water, preferably less than 1 gram per 100 grams of water.

[0023] According to an even more preferred embodiment we make tablets, wherein the relative amount of the water-swelling clay and the water-insoluble inorganic material , other than zeolites in the disintegrant granule is in the weight ratio range of the water-swelling clay: water-insoluble inorganic material of from 0 to 2:1 by weight.

[0024] Where a composition according to the invention comprises an alkali metal aluminosilicate as a detergency builder, it is preferred that at least a part of the aluminosilicate constituent of the cleaning composition is employed as the inorganic material used in the formation of the disintegrant granules.

[0025] The tablets will in general contain 0.5 to 22 wt % (on total tablet) of the STS and/or SCS. If the STS and/or SCS is present in the matrix material the amount thereof will be 0.5 to 5 wt % on total tablet, if this compound is present in the granules the amount will be 0.15 to 50 wt %.

[0026] Generally the tablets according to the invention will contain from 1% to 20% by weight of the disintegrant granules based on the total weight of the compositions, preferably 2 to 15%, more preferably 3 to 10%, e.g. 4 to 8% by weight. If the granules are included to aid dissolution only of the tablets rather than disintegration, then the amount of the granules in the tablets could be as low as 1% by weight.

[0027] The water-swellable agent preferably has an average primary particle size (as measured by a statistical set of sieves) of up to about 600um, but, conveniently, has an average primary particle size of no more than 200 um, preferably no more than 100um.

[0028] When the water insoluble inorganic material in the disintegrant granule is a zeolite it is preferred that the disintegrant granules have a mean particle size (by sieve analysis) in the range 700 to 1200 micrometers. When the water insoluble inorganic material is silica it is preferred that the disintegrant granules have a mean particle size in the range 250 to 700 micrometers.

[0029] The water swellable agent used in the granules or tablets according to the invention preferably has a water-swelling capacity of at least 5 cm^3/gram, preferably 10cm^3/gram and more preferably 20cm^3/gram as determined in the test described under "determination of water swelling capacity.

[0030] The tablets according to the invention also can contain disintegrating promoting agents. These are well known in this area and can be selected from compounds with a high water solubility , from certain tripolyphosphates (either in phase I or partially hydrated).Therefore we have a preference for tablets that comprise water-soluble disintegration-promoting particles containing at least 40%, by weight of the particles, of one or more materials selected from the group consisting of;

i) compounds with water-solubility exceeding 50 grams per 100 grams water at 20°C, and
ii) sodium tripolyphosphate containing at least 50% of its own weight of the phase I anhydrous form, and
iii) sodium tripolyphosphate which is partially hydrated so as to contain water of hydration in an amount which is at least 0.5% by weight of the sodium tripolyphosphate in the particles.

[0031] The compounds with the high water solubility are preferably selected from the group consisting of sodium acetate, in particular the trihydrate thereof, sodium citrate dihydrate and urea. These components are used in amounts of 5 to 25 wt % based on total composition.
Preferred tablets comprise 20 to 45% by weight of the water-swelling clay, 45 to 70% by weight of the water-insoluble inorganic material and 3 to 9% by weight of the water-swellable agent based on the weight of the disintegrant granule.

[0032] The disintegrant granules comprising the inorganic material and the water-swellable agent may be prepared by any of the methods that will be known to those skilled in the art, e.g. by blending the dry ingredients in a mixer (such as a Pek mixer available from George Tweedy & Co of Preston - 281b S.A. Machine) and compacting on a roller compactor (Alexanderwerk WP50 - manufactured by Alexanderwerk AG, D 5630 Remschied 1, Germany).

[0033] A typical small-scale preparative method is described in EP application 01200407.3

Surfactant Compounds

[0034] The compositions of this invention contain one or more non-soap surfactants. In a fabric washing composition, these preferably provide from 5 to 50% by weight of the composition of the tablet or region thereof, more preferably from 8 or 9% by weight of the composition up to 35% or 40% by weight. If the tablet is composed of more than one discrete region, then these preferred amounts of surfactant may apply to the tablet as a whole.

[0035] The organic surfactant may be present as a component in granulated particles in an amount between 10 and 90% by weight of the particles, more preferably 15 to 50% by weight based on the total weight of the granulated particles. All the surfactant in the composition may be contained within these particles. The surfactant may be anionic (soap or non-soap), cationic, zwitterionic, amphoteric, nonionic or a combination of these.

[0036] Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkyl benzene sulphonates, particularly sodium linear alkyl benzene sulphonates having an alkyl chain length of C_8-C_{15}; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; fatty acid ester sulphonates and primary alcohol sulphates.

[0037] It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rape seed oil.

[0038] 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.

[0039] Amphoteric surfactants which may be used jointly with anionic or nonionic surfactants or both and include amphopropionates

[0040] Cationic surfactants may possibly be used. These frequently have a quaternised nitrogen atom in a polar head group and an attached hydrocarbon group of sufficient length to be hydrophobic.

Detergency Builder

[0041] A composition which is compacted to form tablets or tablet regions typically contains a detergency builder which serves to remove or sequester calcium and/or magnesium ions in the water. Thus the builder acts as a water softener. In detergent tablets the amount of builder is from 5% to 80%, more usually 10% or 15% to 40%, 55% or 60% by weight of the tablet.

[0042] The detergency builder may be present in granulated particles in an amount of from 20 to 80% by weight, more preferably 20%, 25% or 30 to 60% by weight.

[0043] Detergency builders may be provided wholly by water soluble materials, or may be provided in large part or even entirely by water-insoluble material with water-softening properties.

[0044] Alkali metal aluminosilicates are strongly favoured as environmentally acceptable detergency builders for fabric washing, and are preferred in this invention. Alkali metal (preferably sodium) aluminosilicates may be either crystalline or amorphous or mixtures thereof, having the general formula:

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

[0045] These materials contain some bound water (indicated as xH_2O) and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

[0046] Conceivably a detergency builder could be a layered sodium silicate as described in US 4 664 839.

[0047] The less preferred category of water-soluble phosphorus-containing inorganic softeners includes the alkali-metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates.

[0048] Non-phosphorus water-soluble detergency builders may be organic or inorganic. Inorganics that may be present include alkali metal (generally sodium) carbonate; while organics include polycarboxylate polymers and monomeric polycarboxylates.

[0049] Tablet compositions preferably include polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers which have some function as water-softening agents and also inhibit unwanted deposition onto fabric from the wash liquor.

[0050] Where the tablet contains water soluble builder it is preferably present in an amount of from 10 to 80% by weight based on the total weight of the tablet or region thereof. Where the tablet contains water in-soluble builder it is preferably present in an amount of from 5 to 80% by weight based on the total weight of the tablet or region thereof.

[0051] For the avoidance of doubt, where a tablet is heterogenous, the percentage ranges for the components referred to herein may apply to the overall composition of the tablet, as well as to at least one region of the tablet.

Optional bleach system

[0052] Tabletted compositions 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 composition of the tablet or region thereof.

[0053] Bleach activators, also referred to as bleach precursors, have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example, tetraacetylethylene diamine (TAED), now in widespread commercial use in conjunction with sodium perborate and sodium percarbonate; and perbenzoic acid precursors.

[0054] A bleach system may also include a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

Enzymes

[0055] Tablets according to invention can also comprise one or more detergency enzymes. Preferably the enzyme is selected from amylase, protease, cellulase, lipase and mixtures thereof. The aforementioned enzymes are designed to remove a variety of soils and stains from fabrics.

Optional other ingredients

[0056] The 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.

[0057] An antifoam material is advantageously included if organic surfactant is present, 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 266 863A (Unilever).

[0058] It may also be desirable that a 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.

Bulk density of non-compacted detergent powder & granulation

[0059] While the starting particulate composition from which the tablets are produced may in principle have any bulk density, the present invention may be especially relevant to tablets of detergent compositions 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, compared with a tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

[0060] Thus the starting particulate composition may suitably have a bulk density of at least 400 g/litre, preferably at least 500 g/litre, and possibly at least 600 g/litre.

[0061] Granular detergent compositions of high bulk density prepared by granulation and densification in a high-speed mixer/granulator, as described and claimed in EP-A-340 013 (Unilever), EP-A-352 135 (Unilever), and EP-A-425 277 (Unilever), or by the continuous granulation/densification processes described and claimed in EP-A-367 339 (Unilever) and EP-A-390 251 (Unilever), are inherently suitable for use in the present invention.

[0062] Another particularly suitable process for the preparation of a high-bulk density detergent powder is described in WO-A-98/11193 (Unilever).

Particle size control

[0063] Particle sizes can be controlled in the manufacturing process of any particles included in the composition. Oversized particles are usually removed by sieving (for example by a Mogensen screen) at the end of the production process, followed by milling and recycling of the removed oversized fraction. Undersized particles can also be removed by sieving, or if the manufacturing process employs a fluidised bed then undersized particles may be entrained in the air stream and subsequently recovered from it for recycling to the granulation stage.

[0064] Materials which are mixed with the granulated particles may also comply with these requirements concerning particle size. These materials (post-added) typically comprise from 5% - 60% by weight of the total weight of the final composition, more usually 35 to 55% by weight.

Tabletting

[0065] Tabletting entails compaction of a particulate composition. A variety of tabletting machinery is known, and can be used. Generally it will function by stamping a quantity of the particulate composition which is confined in a die. Tabletting 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 tabletting at a temperature which is above ambient, the particulate composition is preferably supplied to the tabletting machinery at an elevated temperature. This will of course supply heat to the tabletting machinery, but the machinery may be heated in some other way also. 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.

[0066] The size of a tablet will suitably range from 10 to 160 grams, preferably from 15 to 60 grams, 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.

[0067] A very suitable process for making a tablet of compacted particulate detergent composition comprising non-soap surfactant and detergency builder, the process comprising mixing disintegrant granules comprising a water-swelling clay, a water-insoluble inorganic material and a water swellable agent and optionally STS and/or SCS with the other constituents of the detergent composition, optionally comprising STS and/or SCS to produce a particulate detergent composition comprising STS and/or SCS, placing a quantity of the resultant particulate detergent composition within a mould, and compacting the composition within the mould to produce the tablet.

Tablet testing - disintegration

[0068] The break-up , dispersion and dissolution of tablets was measured by a test procedure in which a tablet is placed on a plastic sieve with 2 mm size which was immersed in 9 litres of tap water at ambient temperature of 22°C and rotated at 200 rpm. The water conductivity was monitored until it reached a constant value.

[0069] The time for break up and dispersion of the tablets was the time as the time (T90) for change in water conductivity to reach 90 % of its final magnitude. This was also confirmed by visual obsrvation of the material remaining on the rotating sieve.

Tablet Strength

[0070] The strength of the tablets, in their dry state as made on the compaction press, can be determined according to their diametrical fracture stress DFS, which is calculated from the equation:

$$DFS = \frac{2F_{max}}{\pi Dt}$$

where DFS is the diametrical fracture stress in Pascals, F_{max} is the applied load in Newtons to cause fracture, D is the tablet diameter in metres and t is the tablet thickness in metres.

The test is carried out using an Instron type universal testing instrument to apply compressive force on a tablet diameter (i.e. perpendicular to the axis of a cylindrical tablet). It is preferred that tablets have a DFS of at least 20 kPa more preferably at least 25 kPa, such as 30 kPa or above.

[0071] It is intended that the tablets, when used as fabric washing tablets in an automatic washing machine, may be added either to the powder dispensing drawer, or, directly in to the washing drum. This may occur either manually or automatically.

Determination of the water-swelling capacity

[0072] To demonstrate the water-swelling capacity of the water-swellable agent, 19.6 grams of the agent or carrier was blended with 0.4 grams of ultramarine pigment and compressed into a tablet using a laboratory tablet press at about 250 MPa to give a tablet of 32 mm diameter. This was crushed and sieved to give granules of particle size 500 - 1000 um. A glass tube, 33 mm in internal diameter and about 30 cm long with a sintered porous glass disk (porosity 1) fitted at one end was immersed upright, with said one end lowermost, in a large beaker of water (at 25°C) so that the water level rose to about 14 cm above the sintered glass. 1 gram of the granules was added to the tube and allowed to settle on the sintered glass disc.

With this arrangement water has access to the granules from both above and below. The granules immediately began to swell, forming a jelly-like mass. The ultramarine pigment imparted a blue colour to the mass making it easy to see the end and to record its height. The height of the swelling mass was recorded at intervals and showed an initial rapid rise followed by a level off after about 20-30 minutes. From the diameter of the tube, the volume of the swollen mass can be calculated.
The result was expressed as cm^3/g of the water swellable agent after 20 minutes.

EXAMPLES

PREPARATION OF THE TABLETS

[0073] Base powder with the composition as mentioned in table 1 was produced via a non-tower route as described in e.g. PCT/EP 02/00945.

[0074] A post dose mix with the composition as mentioned in table 1 was prepared by dosing the individual components into a Halvor Forberg mixer type F500/538 and mixing for 90 seconds.

[0075] The powder for tableting (= base powder + post dose mix + acetate + disintegrant) was prepared by mixing 5 kg material for 1 min in a concrete mixer.

[0076] The powder so obtained was tableted to a strength of DFS 30 kPa using a Kilian SP 300/01-SP-3 single punch tableting machine. Tableting speed was 15 to 20 tablets per minute. The blender speed was 40 RPM and the dwell time was set to zero.

TABLE 1

BASE POWDER FORMULATION:	
Na-LAS	21.22
Nonionic 7 EO	8.50
Soap	1.62
Zeolite A24 (anh.)	43.89
Carbonate	8.83
SCMC (100 %)	1.33
Na-acetate (anhydrous or 3aq)	8.83
Moisture, Salts, NDOM	5.78
TOTAL	100.00

POST-DOSE POWDER:	
Percarbonate	50.12
TAED granulate	14.83
Dequest 2047	2.72
Granular sodium disilicate	9.18
Sodium citrate 2aq	9.18
Dequest 2016	2.22
Antifoam granule	6.59
Fluorescer granule	4.40
Perfume	0.78
TOTAL	100.00

Disintegrant granule:	SID 883	SID 884	SID 885	SID 863	SID 867	SID 892	SID 893	SID 894
Zeolite A24 ex. Ineos Silicas	92.5	82.5	77.5	5.5	5.0	82.5	77.5	77.5
Zeolite A28 ex. Ineos Silicas				49.5	45.5			
Bentonite clay RT 6091 ex. Volclay				37.5	37.1			
Cross-linked SCMC	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5
Aquasorb A500 ex. Hercules								
Sodium Toluene Sulphonate Eltesol STS 90 powder ex. Huntsman		10.0	15.0		5.0			7.5
Na-acetate anhydrous ex. Merck						10.0	15.0	7.5
TOTAL	100	100	100	100	100	100	100	100

[0077] The tablets obtained were tested and the results of these tests are given in table 2.

TABLE 2

	REFERENCE : disintegrant without STS, no STS post-dosed	disintegrant without STS, 2 % STS post-dosed	disintegrant with10 % STS, no STS post-dosed	disintegrant with10 % STS, 2 % STS post-dosed	disintegrant with15 % STS, no STS post-dosed	disintegrant with15 % STS, 2 % STS post-dosed	REFERENCE : bentonite containing disintegrant without STS, no STS post-dosed	bentonite containing disintegrant without STS, 2 % STS post-dosed	bentonite containing disintegrant with 5 % STS, no STS post-dosed
TOTAL FORMULATION	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
% base powder	51.5	50.3	51.5	50.3	51.5	50.3	51.5	50.3	51.5
% post-dose mix	33.5	32.7	33.5	32.7	33.5	32.7	33.5	32.7	33.5
Na-acetate trihydrate /zeolite A24 99:1 mix	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00
additive	0.00	2.00	0.00	2.00	0.00	2.00	0.00	2.00	0.00
type		Eltesol STS 90 powder ex. Huntsman		Eltesol STS 90 powder ex. Huntsman			Eltesol STS 90 powder ex. Huntsman	Eltesol STS 90 powder ex. Huntsman	
Tablet disintegrant	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00
type	SID 883	SID 883	SID 884	SID 884	SID 885	SID 885	SID 863	SID 863	SID 867
TOTAL	15.00	17.00	15.00	17.00	15.00	17.00	15.00	17.00	15.00
initial tablet strength (kPa)	29.0	28.4	27.6	26.5	27.2	28.3	30.5	32.2	31.1
residual strength after 28 days 37°C closed storage (kPa)	12.4	27.2	13.2	20.8	13.3	25.1	10.1	21.9	16.8
residual strength after 28 days 37°C closed storage (% vs. initial)	43	96	48	78	49	89	33	68	54
improvement vs. no additive (ratios residual strength). Additive is considered to be effective if the residual strength is at least 20 % higher than same tablet without additive		2.2	1.1	1.8	1.1	2.1	0.8	1.6	1.3

TABLE 2 (cont.)

	bentonite containing disintegrant with 5 % STS, 2 % STS post-dosed	disintegrant with 10 % anhydrous acetate, no STS post-dosed	disintegrant with 10 % anhydrous acetate, 2 % STS post-dosed	disintegrant with 15 % anhydrous acetate, no STS post-dosed	disintegrant with 15 % anhydrous acetate, 2 % STS post-dosed	disintegrant with 7.5 % anhydrous acetate and 7.5 % STS, no STS post-dosed
TOTAL FORMULATION	**10**	**11**	**12**	**13**	**14**	**15**
% base powder	50.3	51.5	50.3	51.5	50.3	51.5
% post-dose mix	32.7	33.5	32.7	33.5	32.7	33.5
Na-acetate trihydrate / zeolite A24 99:1 mix	12.00	12.00	12.00	12.00	12.00	12.00
additive	2.00	0.00	2.00	0.00	2.00	0.00
type	Eltesol STS 90 powder ex. Huntsman		Eltesol STS 90 powder ex. Huntsman		Eltesol STS 90 powder ex. Huntsman	
Tablet disintegrant	3.00	3.00	3.00	3.00	3.00	3.00
type	SID 867	SID 892	SID 892	SID 893	SID 893	SID 894
TOTAL	17.00	15.00	17.00	15.00	17.00	15.00
initial tablet strength (kPa)	30.9	23.3	26.1	23.5	24.8	22.0
residual strength after 28 days 37°C closed storage (kPa)	25.9	11.2	21.1	11.3	20.3	12.6
residual strength after 28 days 37°C closed storage (% vs. initial)	84	48	81	48	82	57
improvement vs. no additive (ratios residual strength). Additive is considered to be effective if the residual strength is at least 20 % higher than same tablet without additive	2.0	1.1	1.9	1.1	1.9	1.3

TABLE 2 (cont.)

	disintegrant with 7.5 % anhydrous acetate and 7.5 % STS, 2 % STS post-dosed	REFERENCE : disintegrant without STS, no STS post-dosed	disintegrant without additive, 2 % additive post-dosed	disintegrant without additive, 2 % additive post-dosed	disintegrant without additive, 2 % additive post-dosed
TOTAL FORMULATION	**16**	**17**	**18**	**19**	**20**
% base powder	50.3	51.5	50.3	50.3	50.3
% post-dose mix	32.7	33.5	32.7	32.7	32.7
Na-acetate trihydrate / zeolite A24 99:1 mix	12.00	12.00	12.00	12.00	12.00
additive	2.00	0.00	2.00	2.00	2.00
type	Eltesol STS 90 powder ex. Huntsman		Eltesol STS 90 powder ex. Huntsman	terephthalic acid	Na-naphtalene sulphonate
Tablet disintegrant	3.00	3.00	3.00	3.00	3.00
type	SID 894	SID 883	SID 883	SID 883	SID 883
TOTAL	17.00	15.00	17.00	17.00	17.00
initial tablet strength (kPa)	27.4	28.3	30.2	33.4	29.3
residual strength after 28 days 37°C closed storage (kPa)	24.5	10.8	22.2	11.9	11.9
residual strength after 28 days 37°C closed storage (% vs. initial)	89	38	74	36	41
improvement vs. no additive (ratios residual strength). Additive is considered to be effective if the residual strength is at least 20 % higher than same tablet without additive	2.1		1.9	0.9	1.1

TABLE 2(cont.)

	disintegrant without additive, 2 % additive post-dosed	disintegrant without additive, 2 % additive post-dosed	disintegrant without additive, 2 % additive post-dosed	disintegrant without additive, 2 % additive post-dosed	disintegrant without additive, 2 % additive post-dosed
TOTAL FORMULATION	**21**	**22**	**23**	**24**	**25**
% base powder	50.3	50.3	50.3	50.3	50.3
% post-dose mix	32.7	32.7	32.7	32.7	32.7
Na-acetate trihydrate / zeolite A24 99:1 mix	12.00	12.00	12.00	12.00	12.00
additive	2.00	2.00	2.00	2.00	2.00
type	2,6 dimethyl naphtalene	boric acid	sorbitol	SXS	SCS
Tablet disintegrant	3.00	3.00	3.00	3.00	3.00
type	SID 883	SID 883	SID 883	SID 883	SID 883
TOTAL	17.00	17.00	17.00	17.00	17.00
initial tablet strength (kPa)	30.5	29.4	28.9	29.9	31.6
residual strength after 28 days 37°C closed storage (kPa)	11.0	12.2	8.2	12.9	17.6
residual strength after 28 days 37°C closed storage (% vs. initial)	36	41	28	43	56
improvement vs. no additive (ratios residual strength). Additive is considered to be effective if the residual strength is at least 20 % higher than same tablet without additive	0.9	1.1	0.7	1.1	1.5

Claims

1. A tablet of compacted particulate detergent composition comprising non-soap surfactant and detergency builder, wherein the tablet or a discrete region thereof comprises a matrix material and disintegrant granules comprising a water-insoluble inorganic material and a water-swellable agent and a water-swelling clay, while either the granules or the matrix material or both comprise an agent, selected from the group consisting of STS and SCS.

2. A tablet according to claim 1 wherein the water-swellable agent in its anhydrous state, comprises 1 to 20% by weight of the combined weight of the water-insoluble inorganic material, the STS and/or SCS and the water-swellable agent and the water-swelling clay.

3. A tablet according to claim 1 wherein the water swelling clay is a bentonite clay.

4. A tablet according to claim 1 wherein the water-insoluble inorganic material is silica, a material containing at least 70% silica by weight or a zeolite.

5. A tablet according to claim 4 wherein the zeolite is a zeolite P having a water content in the range of from 9 to 12% by weight.

6. A tablet according to claim 1 wherein the relative amount of the water-swelling clay and the water-insoluble inorganic material , other than zeolites , in the disintegrant granule is in the weight ratio range of from 0 to 2:1 by weight of the water-swelling clay: water-insoluble inorganic material.

7. A tablet according to claim 1 wherein the water-swellable agent is selected from the group consisting of cellulose, cross-linked cellulose, carboxymethyl cellulose, sodium carboxymethyl cellulose, cross-linked sodium carboxymethyl cellulose, pre-gelatinised starch, cross-linked starch and cross-linked polyvinyl pyrrolidone.

8. A tablet according to claim 1 wherein the water-swellable agent comprises in its anhydrous state 1 to 10% by weight of the combined weight of water-insoluble inorganic material, the STS and/or SCS and the water-swellable agent and the water swelling clay.

9. A tablet according to claim 1 wherein the water-swellable agent in the disintegrant granule is present in an amount of less than 2% by weight based on the total weight of the tablet composition.

10. A tablet according to claim 1 wherein the STS and/or SCS is present in 0.5 to 22 wt % on total tablet.

11. A tablet according to claim 1 wherein the STS and/or SCS is present in the matrix material in an amount of 0.5 to 5 wt % on total tablet.

12. A tablet according to claim 1 wherein the STS and/or SCS is present in the granules in an amount of 0.15 to 50 wt % on total tablet.

13. A tablet according to claim 1 wherein the water swellable agent has an average primary particle size of up to 600 um.

14. A tablet according to claim 1 wherein the tablet contains from 1 to 20% by weight of the disintegrant granules based on the total weight of the tablet composition.

15. A tablet according to claim 1 wherein the disintegrant granules contain a zeolite as water insoluble inorganic material and have a mean particle size in the range of from 700 to 1200 micrometers.

16. A tablet according to claim 1 which further comprises water-soluble disintegration-promoting particles containing at least 40%, by weight of the particles, of one or more materials selected from the group consisting of;

    i) compounds with water-solubility exceeding 50 grams per 100 grams water at 20°C, and
    ii) sodium tripolyphosphate containing at least 50% of its own weight of the phase I anhydrous form, and
    iii) sodium tripolyphosphate which is partially hydrated so as to contain water of hydration in an amount which is at least 0.5% by weight of the sodium tripolyphosphate in the particles.

17. A tablet according to claim 16 wherein the compounds with water-solubility exceeding 50 grams per 100 grams water at 20°C are selected from sodium acetate, sodium citrate dihydrate or urea.

18. A tablet according to claim 17 wherein the sodium acetate comprises sodium acetate trihydrate.

19. A tablet according to claim 16 wherein the water-soluble disintegration-promoting particles are present in an amount of from 5% to 25% by weight based on the total weight of the composition.

20. A process for making a tablet of compacted particulate detergent composition comprising non-soap surfactant and detergency builder, the process comprising mixing disintegrant granules comprising a water-swelling clay, a water-insoluble inorganic material and a water swellable agent and optionally the STS and/or SCS with the other con-

stituents of the detergent composition, optionally comprising the STS and/or SCS to produce a particulate detergent composition comprising STS and/or SCS, placing a quantity of the resultant particulate detergent composition within a mould, and compacting the composition within the mould to produce the tablet.

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EUROPEAN SEARCH REPORT

Application Number
EP 03 07 7710

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	4 December 2003	Richards, M

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