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(54) **DETERGENT COMPOSITIONS**

WASCHMITTELZUSAMMENSETZUNGEN

COMPOSITIONS DETERGENTES

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Description

[0001] This invention relates to detergent compositions in the form of tablets for use in fabric washing.

[0002] Detergent compositions in tablet form have been described in, for example, GB 911204 (Unilever), US 3953350 (Kao), JP 60-015500A (Lion), and EP-A-711827 (Unilever) and are sold commercially in Spain. Tablets have several advantages over powdered products: they do not require measuring and are thus easier to handle and dispense into the washload, and they are more compact, hence facilitating more economical storage.

[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. It has not proved simple to achieve both properties simultaneously. As more pressure is used when a tablet is compacted, so the tablet density and strength rise, but the speed of disintegration/dissolution when the tablet comes into contact with wash water goes down.

[0004] During the period from about 1960 to 1970 there was considerable research activity in connection with tablets for use in fabric washing. A number of patents were published by major detergent manufacturers. Detergent tablets were sold commercially in USA and some European countries.

[0005] However, tablets disappeared from the market place in nearly all countries (Spain is the apparent exception) even though tablets have apparent advantages and have become known as a product form for machine dishwashing compositions which are characterised by a low content of organic surfactant.

[0006] 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.

[0007] The compression pressure used in the Examples of this document was from 180 to 300 psi (approximately 1.2 to 2.1 MPa). Elsewhere in the document it is proposed that the pressure should not exceed 350 psi (approximately 2.5 MPa) to avoid slow disintegration encountered with higher pressures.

[0008] A number of proposals have been put forward as ways to improve the compromise between conflicting desiderata, but there still remains a desire to improve tablet strength without loss of speed of disintegration and vice versa.

[0009] Some documents have proposed surface treatments or coatings to enhance tablet strength. For instance US-A-3451928 (Colgate) stated that the problem of strength versus speed of dissolution remained unsolved, and proposed a treatment of spraying on water, followed by flash heating.

US-A-3324038 (Procter) proposed the application of a coating containing urea.

[0010] It is known to include 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. Urea has a very high solubility in water exceeding 100gms per 100ml water at 20°C. EP-A-711827 teaches the use of sodium citrate for the same purpose.

[0011] Detergent compositions, including tablet compositions, frequently contain a mixture of anionic and nonionic organic surfactants. It is often desirable to include both of these types of surfactant, for performance of the composition when washing fabrics.

[0012] We have now found that an improved compromise between tablet strength and speed of disintegration can be achieved by following certain principles concerning tablet formulation and manufacture.

[0013] At the same time it is possible to incorporate materials which are desired to give good washing performance, and it is possible to formulate component ingredients of the tablet so that they are satisfactory in handling during tablet manufacture.

[0014] Also, we have found that two different measures of tablet strength are relevant to properties observed by a consumer. Force to cause fracture is a direct assessment of strength and indicates 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.

[0015] Both properties are relevant to consumers' perception of tablets: consumers will want tablets to be strong enough to handle, to reach them intact, and to disintegrate quickly and fully at the time of use. By following this invention's principles as to formulation, it is possible to achieve a good combination of these properties.

[0016] In this invention, we have found it desirable to incorporate most (if not all) of the surfactants in particles which constitute a substantial part but by no means all the composition of a tablet. The organic surfactant in these particles provides a substantial part, but again by no means all, of their weight.

[0017] Secondly, it is desirable that the surfactant is a mixture of non-soap anionic and nonionic detergent surfactants (preferably accompanied by soap) where both are present in significant amounts, but anionic is in the majority. Thirdly, the balance of the composition should then contain material, other than organic surfactant, which is water soluble. This approach to formulation is applied to the composition of a whole tablet.

[0018] So, according to a first aspect of this invention, there is provided a detergent tablet of compressed particulate composition wherein the tablet comprises organic surfactant and water-soluble detergency builder, characterised in that the tablet contains from 30 to 65% by weight (of the tablet) of particles which contain from 25 to 80% by weight

(of these particles) of water-soluble detergency builder and from 20 to 50% by weight (of these particles) of non-soap organic surfactant which is anionic and nonionic surfactants in a ratio from 5:1 to 1.5:1 and in that in addition to said particles the tablet contains at least 15% by weight (of the tablet) of substance which is other than soap or organic surfactant and which has a solubility in water of at least 10gm/litre at 20°C.

[0019] We believe that concentrating most or all of the surfactant into surfactant-rich particles, and using a substantial proportion of anionic surfactant is beneficial in providing tablets which have both strength and elasticity, while allowing the remainder of the tablet composition to contain a substantial proportion of water-soluble material which assists disintegration of the tablets at the time of use.

[0020] It is not necessary to include surfactant as a binder material in the part of the composition outside the surfactant-rich particles. Excluding it from this part of the composition is advantageous, to avoid interference with the prompt dissolution of this part of the composition.

[0021] So, it is preferred that the weight of the non-soap anionic surfactant in the particles is at least 1.7 times the weight of the nonionic surfactant in them. More preferably, this weight ratio of anionic surfactant to nonionic surfactant lies in a range from 2:1 up to 5:1, and more preferably from 2:1 to 4:1. Preferably these particles contain at least 80% by weight better at least 90% or even 95% of all the organic surfactant (including any soap) in the tablet.

[0022] The water-soluble material which is present in the composition, externally to the surfactant-rich particles, preferably comprises from 15 to 40% (better 16 or even 25% up to 35%) by weight (of the tablet) of one or more materials selected from

- compounds with a water-solubility exceeding 50 grams per 100 grams water;
- sodium tripolyphosphate containing at least 50% of its own weight of the phase I anhydrous form, and preferably partially hydrated so as to contain water of hydration in an amount which is at least 1% by weight of the sodium tripolyphosphate;
- mixtures thereof.

[0023] It is strongly preferred that the water-soluble material which is present in the composition, externally to the surfactant-rich particles, is present as particles which are substantially free of surfactant, i.e. contain no more than 5% of their own weight of organic surfactant.

[0024] A preferred form of this invention provides a detergent tablet of compressed particulate composition containing

(i) from 35 to 60 wt% (and probably from 41 to 53, 56 or 60wt%) of particles which contain non-soap anionic surfactant, nonionic surfactant and water-soluble detergency builder,
(ii) from 15 to 40 wt%(and probably from 16 or 1.7 to 35 wt%) of particles which are substantially free of surfactant, i.e. contain at least 95% of their own weight of water soluble material but contain no more than 5% of their own weight of organic surfactant, and
(iii) from 0 to 50 wt% of further particulate ingredients,

wherein the first said particles (i) contain at least 20% preferably at least 24% of their own weight of non-soap surfactant and the weight of anionic surfactant therein is from 1.5 to 5 times the weight of nonionic surfactant therein.

[0025] In another aspect, this invention provides a detergent tablet of compressed particulate composition wherein the tablet comprises particles which contain non-soap anionic surfactant, nonionic surfactant, preferably soap and other water-soluble ingredients, characterised in that the particles contain at least 20 wt% in total of the anionic and nonionic surfactants and in that a test tablet consisting of the said non-soap anionic surfactant, nonionic surfactant, and any soap in the same proportions, together with 15% by weight moisture has a breaking strength as herein defined of at least 0.4 MPa and a modulus as herein defined of not more than 10 MPa preferably not more than 8 MPa.

[0026] In a yet further aspect, this invention provides the use of particles containing a mixture of non-soap anionic and nonionic surfactants and detergency builder, where the concentration by weight of non-soap anionic surfactant is at least 1.5 times as great as the concentration by weight of nonionic surfactant and the total concentration of these surfactants is at least 20 wt% of the particles to provide improvements in tablet strength and elasticity versus speed of disintegration.

[0027] The tablet of the invention is homogeneous. 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 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.

Drawings

[0028] The accompanying drawings diagrammatically illustrate the testing of a cylindrical tablet:

Fig 1a shows a tablet when first contacted by the platens of a materials testing machine,
Fig 1b shows the tablet at the point of failure,
Fig 2 diagrammatically illustrates the form of a graph obtained during testing.

Tablet testing

[0029] We have tested the speed of disintegration of tablets by means of a test procedure in which a tablet was placed on a plastic sieve with 2mm mesh size which is immersed in 9 litres of demineralised water at ambient temperature of 20°C. The water conductivity is monitored until it reached a constant value. The time for dissolution of the tablets is taken as the time (T_{90}) for change in the water conductivity to reach 90% of its final magnitude.

[0030] We have tested tablet strength by a procedure illustrated by the accompanying drawings in which a cylindrical tablet 10 is compressed radially between the platens 12,14 of a materials testing machine until the tablet fractures. At the starting position shown in Fig 1a, the platens 12, 14 contact the tablet but do not apply force to it. Force is applied, as indicated by the arrows 16 to compress the tablet. The testing machine measures the applied force (F), and also the displacement (x) of the platens towards each other as the tablet is compressed. The distance (y) between the platens before force is applied, which is the diameter of the tablet, is also known. At failure, illustrated in Fig 1b the tablet cracks (eg as shown at 18) and the applied force needed to maintain the displacement drops. Measurement is discontinued when the applied force needed to maintain the displacement has dropped by 25% from its maximum value (indicated as point D in Fig 2 below).

[0031] A graph of force (F) against displacement (x) has the form illustrated by Fig 2. The maximum force is the force at failure (F_f). From this measurement of force a test parameter called diametral fracture stress, which we have used in the past, can be calculated using the equation

$$\sigma = \frac{2F_f}{\pi Dt}$$

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.

[0032] The force at failure divided by the area of a diametral plane through the tablet (approximately the area of the crack 18) is the breaking strength, with units of Pascals.

[0033] The break energy is the area under the graph of force against displacement, up to the point of break. It is shown shaded in Fig 2 and is given by the equation:

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

where 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.

[0034] The displacement at failure relative to the tablet diameter is the relative displacement x_f/y.

[0035] Breaking strength divided by relative displacement is a modulus, whose value is inverse to tablet elasticity.

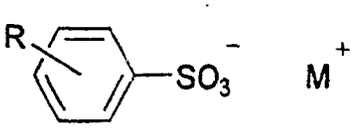
Materials and other features

[0036] Materials which may be used in tablets of this invention will now be discussed in more detail.

Anionic Surfactant Compounds

[0037] Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. The anionic surfactant

may comprise, wholly or predominantly, linear alkyl benzene sulphonate of the formula



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

[0038] Primary alkyl sulphate having the formula

$$ROSO_3^- M^+$$

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

[0039] 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 surfactant and may provide 75 to 100wt% of any anionic non-soap surfactant in the composition.

[0040] Examples of other non-soap anionic surfactants include olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

[0041] One or more soaps of fatty acids may also be included in addition to the required non-soap anionic surfactant. Examples are sodium soaps derived from the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil. These may be formed by adding fatty acid and a base such as sodium carbonate to a slurry which is spray-dried to form the surfactant-rich base particles.

Nonionic surfactant compounds

[0042] Nonionic surfactant compounds 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.

[0043] Specific nonionic surfactant compounds are alkyl (C_{8-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-diamine.

[0044] Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{9-11} and C_{12-15} primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.

Detergency Builder

[0045] The composition which is compacted to form tablets includes water-soluble detergency builder.

[0046] Water-soluble phosphorous-containing inorganic detergency builders include the alkali-metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates. Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, orthophosphates and hexametaphosphates.

[0047] 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.

[0048] 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.

Proportions

[0049] Generally, a tablet in accordance with this invention will contain overall from 2 or 5wt% up to 40 or 50wt% non-soap surfactant, and from 5 or 10wt% up to 60 or 80wt% detergency builder.

[0050] The concentration of non-soap anionic surfactant in the tablet will generally be at least one and a half times the concentration of nonionic surfactant. It is preferably at least 3% by weight of the tablet, e.g. from 3wt% up to 30 or 40wt%. The concentration of nonionic surfactant is preferably at least 2% by weight of the tablet, such as 2 to 15wt%

of the tablet.

[0051] The quantity of soap in the tablet is preferably from 0.1 or 0.2 up to 1% or 2% by weight of the tablet. Higher proportions such as up to 4% are less preferred.

[0052] In accordance with this invention, anionic non-soap surfactant, nonionic surfactant water-soluble detergency builder and other materials which preferably include soap are made into particles such that the non-soap surfactant provides from 20 to 50% of the weight of these particles. Preferably the non-soap surfactant provides at least 22% or 24% of the weight of these particles, and more preferably at least 28%, desirably up to 45% of their weight. When soap is present, it is desirably from 0.2 to 2%, and possibly more, up to 3% or 4% by weight of these particles, and in these particles the weight ratio of nonionic detergent to soap is preferably from 5:1 better 10:1 to 30:1.

[0053] Such particles may be made by spray drying, or by a granulation process. Preferably they contain water-soluble detergency builder in an amount which is from 30 to 80% of the weight of these particles, better 30 or 40 up to 60% of the weight of these particles.

[0054] The surfactant mixture used in these particles can be tested mechanically in directly analogous manner to the testing of tablets, discussed above. To do this a mixture of the non-soap surfactants and any soap is made on a small scale, and cast into cylindrical form or some other shape from which a cylinder can be cut. If necessary this is dried to reduce the water content to 15% by weight (approximating to 5% moisture in the particles which contain this surfactant mixture). Next, it is tested on a materials testing machine in the manner described above for testing of tablets. This mechanical testing procedure can also be applied to tablets made from the surfactant-rich particles alone.

[0055] We have found that the effect of anionic surfactant in these particles is to enhance elasticity without much effect on magnitude of the force to cause fracture. Nonionic surfactant tends to have some opposite effect. Soap when present, cooperates with the nonionic surfactant to reduce mobility of the nonionic surfactant and to increase tablet strength (as measured by force to cause failure).

[0056] By using sufficient quantities of anionic non-soap surfactant, nonionic surfactant and preferably soap we have found that it is possible to achieve adequate strength and elasticity of a test tablet which in turn signifies that the same mixture will give tablets with good strength and elasticity.

[0057] Breaking strength is desirably at least 0.04MPa preferably at least 0.05MPa. The modulus is desirably no more than 10 MPa preferably no more than 8 or even 5 MPa.

[0058] We have observed that a mixture of alkylbenzene sulphonate and nonionic surfactant in ratio 1.16:1 gave a modulus of about 15 MPa but when the proportions were changed to 2.2:1 (in accordance with this invention) the modulus dropped dramatically to about 4.0 to 4.5 MPa, indicating greater elasticity, with very little change in force at failure.

Disintegration-promoting particles

[0059] In addition to the required particles containing surfactants and builder, a tablet of this invention contains water-soluble material which serves to promote disintegration. Preferably this is provided as particles which are substantially free of organic surfactant.

[0060] One preferred 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.

[0061] 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.

[0062] 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.

[0063] 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. A further preference is that the 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.

[0064] Suitable material is commercially available. Suppliers include Rhone-Poulenc, France and Albright & Wilson, UK.

[0065] "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.

[0066] Another possibility which can be used instead of tripolyphosphate, or in a mixture with it, is that these disin-

tegration-promoting particles contain at least 50% of their own weight, better at least 80%, of a material which has a solubility in deionised water at 20°C of at least 50 grams per 100 grams of water.

[0067] The said particles may provide material of such solubility in an amount which is at least 7 wt% or 12 wt% of the whole composition of the tablet.

[0068] 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.

[0069] 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	119
Sodium acetate trihydrate	76
Magnesium sulphate $7H_2O$	71
Potassium acetate	>200

[0070] 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

[0071] 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.

Other ingredients

[0072] 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.

[0073] 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 tetraacetylethylene 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.

[0074] 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.

[0075] 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 granules or marumes, optionally with a protective coating, in amount of from about 0.1% to about 3.0% by weight of the tablet.

[0076] 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.

[0077] 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 granules 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.

[0078] It may also be desirable that a detergent tablet of the invention includes an amount of an alkali metal silicate, particularly sodium ortho-, meta- or disilicate. The presence of such alkali metal silicates may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some detergency building. Preferably the surfactant-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 tabletting.

[0079] 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.

[0080] These various other ingredients may be present in the surfactant-rich particles or in the balance of the composition outside them. It is preferred that any bleach is contained in the balance of the composition outside the surfactant-rich particles.

Particle Size and Distribution

[0081] A detergent tablet of this invention is a matrix of compacted particles.

[0082] Preferably the particulate composition has an average particle size before compaction in the range from 200 to 2000 μm, more preferably from 250 to 1400 μm. Fine particles, smaller than 180 μm or 200 μm may be eliminated by sieving before tableting, if desired, although we have observed that this is not always essential.

[0083] While the starting particulate composition may in principle have any bulk density, the present invention is especially relevant to tablets made by compacting powders of relatively high bulk density, because of their greater tendency to exhibit disintegration and dispersion problems. Such tablets have the advantage that, as compared with a tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

[0084] Thus the starting particulate composition may suitably have a bulk density of at least 400 g/litre, preferably at least 550 g/litre, and perhaps at least 600 g/litre.

[0085] Granular detergent compositions of high bulk density prepared by granulation and densification in a high-speed mixer/granulator, as described and claimed in EP 340013A (Unilever), EP 352135A (Unilever), and EP 425277A (Unilever), or by the continuous granulation/densification processes described and claimed in EP 367339A (Unilever) and EP 390251A (Unilever), are inherently suitable for use in the present invention.

Tableting

[0086] Tableting entails compaction of the particulate composition. 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 die.

[0087] 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.

[0088] 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.

[0089] The size of a tablet will suitably range from 10 to 160 grams, preferably from 15 to 60 g, depending on the conditions of intended use, and whether it represents a dose for an average load in a fabric washing or dishwashing machine or a fractional part of such a dose. The tablets may be of any shape. However, for ease of packaging they are preferably blocks of substantially uniform cross-section, such as cylinders or cuboids. The overall density of a tablet preferably lies in a range from 1040 or 1050gm/litre up to 1300gm/litre. The tablet density may well lie in a range up to no more than 1250 or even 1200gm/litre.

Examples

[0090] Tablets for use in fabric washing were made, starting with spray-dried base powders of the following compo-

sitions:

Ingredient	Parts by Weight		
	A	B	C
Sodium linear alkylbenzene sulphonate	6.4	9.6	11.0
C_{13-15} fatty alcohol 7EO	4.3	1.1	2.4
C_{13-15} fatty alcohol 3EO	3.2	3.2	2.3
Sodium tripolyphosphate*	24.3	24.3	18.0
Sodium silicate	6.4	5.9	4.0
Soap	0.3	0.3	0.21
Acrylate/maleate copolymer	1.2	1.2	1.5
Sodium sulphate, moisture and minor ingredients	balance to 60	balance to 55	balance to 45

* Added to the slurry as anhydrous sodium tripolyphosphate containing at least 70% phase II form.

[0091] Particulate compositions were made by mixing this powder with other ingredients as tabulated 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).

[0092] The compositions contained the following percentages by weight:

Ingredient	% by weight		
	Ex 1	Ex2	Ex 3
Base powder A	60		
Base powder B		55	
Base powder C			45
Sodium percarbonate granules	14	15	15
TAED granules	3.4	3.4	3.4
Anti-foam granules	1.2	1.2	3.2
Perfume, enzymes and other minor ingredients	1.4	2.2	3.5
Rhodiaphos HPA3.5 tripolyphosphate	20	20	30
Sodium carbonate	-	3.2	-

[0093] 40g portions of each composition were made into cylindrical tablets of 44 mm diameter, using a Carver hand press, with sufficient applied pressure to produce tablets which dissolved to 90% of complete dissolution within approximately equal times not exceeding four minutes, in water at 20°C. Applied forces in a range from 2 to 4 kN have usually been found to be enough when using this hand press.

[0094] To test dissolution of tablets, a test procedure was used in which a tablet was placed on a plastic sieve with 2mm mesh size which was immersed in 9 litres of demineralised water at ambient temperature of 20°C. The water conductivity was monitored until it reached a constant value. The time for dissolution of the tablets was taken as the time (T_{90}) for change in the water conductivity to reach 90% of its final magnitude.

[0095] The strength of these tablets was measured by compressing them radially, between the platens of a universal materials testing machine until fracture of the tablet occurred.

[0096] The force at fracture was measured and also the displacement at fracture. The break energy, which as explained above is the area under a force-over-displacement graph up to failure, was calculated.

[0097] The following table gives the results obtained for tablets of Examples 2 and 3 embodying the invention, and comparative tablets of Example 1. It is apparent that tablets embodying the invention were stronger although they dissolved in a similar time. Included in the table are mechanical properties of the surfactant mixture used.

	Ex 1	Ex2	Ex 3
Powder features			
active system anionic/nonionic	6/7	9/4	9/4
level of base powder [%]	60	55	45
active level in base [%]	23	25	35
active/tripolyphosphate in base powder	0.57	0.57	0.88
level of disintegrant [%]	20	20	30
Tablet properties			
force at failure [N]	30.5	35.0	69.5
break energy [m joules]	5.8	10.5	20.4
Properties of surfactant mix			
Breaking strength [MPa]	0.06 - 0.08	0.05 - 0.07	
Modulus [MPa]	15	4.0 - 4.5	

Claims

1. A detergent tablet of compressed particulate composition wherein the tablet is homogeneous and comprises organic surfactant and water-soluble detergency builder, **characterised in that** the tablet contains from 30 to 65% by weight of particles which contain from 25 to 80% by weight (of these particles) of water-soluble detergency builder and from 20 to 50% by weight (of these particles) of non-soap organic surfactant which is anionic and nonionic surfactants in a weight ratio from 5:1 to 1.5:1 and **in that** in addition to said particles the tablet contains 15% or more by weight (of the tablet) of material which is other than soap or organic surfactant and which has a solubility in water of at least 10 gm/litre at 20°C.

2. A tablet according to claim 1 wherein the tablet contains the said particles in an amount from 41 to 56% by weight.

3. A tablet according to claim 1 to 2 wherein the weight ratio of anionic surfactants to non-ionic surfactant lies in range from 1.7:1 to 5:1.

4. A tablet according to any one of claims 1 to 3 wherein the weight ratio of non-ionic surfactant to soap in said particles lies in range from 10:1 to 30:1.

5. A tablet according to any one of claims 1 to 4 wherein the amount of non-soap organic surfactant in the said particles is from 22 to 45% by weight of the particles.

6. A tablet according to any one of claims 1 to 5 containing soup in the said particles in an amount for 0.2 to 2% by weight of the particles.

7. A tablet according to any one of claims 1 to 6 herein the said particles contain from 30 to 80% by weight (of the particles) of water-soluble detergency builder which is an alkali metal salt of a condensed phosphate.

8. A tablet according to claim 7 wherein the salt of a condensed phosphate is sodium tripolyphosphate.

9. A tablet according to any one of claims 1 to 8 wherein the water-soluble material present in addition to the said particles comprises 15% or more (by weight of the tablet) in the form of further particles which contain no more than 5% of their own weight of organic surfactant.

10. A tablet according to any one of claims 1 to 9 wherein the water-soluble substance present in addition to the said particles comprises 25 to 35% (by weight of the tablet) of material with a water solubility of at least 50 gm/litre at 20°C, or sodium tripolyphosphate containing more than 50% of its own weight of the phase I anhydrous form, or

a mixture thereof.

11. A tablet according to claim 10 wherein the water-soluble substance present in addition to the said particles comprises 15 to 35% (by weight of the tablet) of sodium tripolyphosphate containing more than 50% of its own weight of the phase I anhydrous form, which is partially hydrated so as to contain from 1 to 4% by weight of water of hydration.

12. A tablet according to any one of claims 1 to 11 wherein said anionic surfactant is a majority of sodium alkyl benzene sulphonate, optionally accompanied by a smaller amount of other anionic surfactant.

13. A tablet according to any one of claims 1 to 12 wherein said non-ionic surfactant is a majority of ethoxylated fatty alcohol, optionally accompanied by a smaller amount of other non-ionic surfactant.

14. A tablet according to any one of claims 1 to 13 which is a formed by compression of a single particulate composition.

15. A process for making a detergent tablet according to any one of claims 1 to 14 which comprises mixing

a) 30 to 65% by weight of particles which contain from 25 to 80% by weight (of these particles) of water-soluble detergency builder and from 20 to 50% by weight (of these particles) of non-soap organic surfactant which is anionic and non-ionic surfactants in a weight ratio from 5:1 to 1.5:1 with
b) 15% or more by weight of material which is other than soap or organic surfactant and which has a solubility in water of at least 10 gm/litre at 20°C, and compacting the mixture into a tablet.

Patentansprüche

1. Detergens-Tablette aus einer komprimierten teilchenförmigen Zusammensetzung, wobei die Tablette homogen ist und organisches Tensid und einen wasserlöslichen Waschkraft-Builder umfasst, **dadurch gekennzeichnet, dass** die Tablette 30 bis 65 Gewichts-% Teilchen enthält, die 25 bis 80 Gewichts-% (dieser Teilchen) an wasserlöslichem Waschkraft-Builder und 20 bis 50 Gewichts-% (dieser Teilchen) an organischem Nicht-Seifen-Tensid, das anionisch ist, und nichtionischen Tensiden in einem Gewichtsverhältnis von 5:1 bis 1,5:1 enthalten, und dass die Tablette zusätzlich zu den Teilchen 15 oder mehr Gewichts-% (der Tablette) an Material enthält, das von einer Seife oder organischem Tensid verschieden ist und das eine Löslichkeit in Wasser von mindestens 10 g/Liter bei 20°C aufweist.

2. Tablette nach Anspruch 1, in der die Tablette die Teilchen in einer Menge von 41 bis 56 Gewichts-% enthält.

3. Tablette nach Anspruch 1 oder 2, in der das Gewichtsverhältnis von anionischen Tensiden zu nichtionischem Tensid im Bereich von 1,7:1 bis 5:1 liegt.

4. Tablette nach irgendeinem der Ansprüche 1 bis 3, in der das Gewichtsverhältnis von nichtionischem Tensid zu Seife in den Teilchen im Bereich von 10:1 bis 30:1 liegt.

5. Tablette nach irgendeinem der Ansprüche 1 bis 4, in der die Menge an organischem Nicht-Seifen-Tensid in den Teilchen 22 bis 45 Gewichts-% der Teilchen beträgt.

6. Tablette nach irgendeinem der Ansprüche 1 bis 5, die Seife in den Teilchen in einer Menge von 0,2 bis 2 Gewichts-% der Teilchen enthält.

7. Tablette nach irgendeinem der Ansprüche 1 bis 6, in der die Teilchen 30 bis 80 Gewichts-% (der Teilchen) wasserlöslichen Waschkraft-Builder enthalten, bei dem es sich um ein Alkalimetallsalz eines kondensierten Phosphats handelt.

8. Tablette nach Anspruch 7, in der das Salz eines kondensierten Phosphats Natriumtripolyphosphat ist.

9. Tablette nach irgendeinem der Ansprüche 1 bis 8, in der das wasserlösliche Material, das zusätzlich zu den Teilchen anwesend ist, 15% oder mehr (bezüglich Gewicht der Tablette) in Form von weiteren Teilchen umfasst, die nicht mehr als 5% ihres eigenen Gewichts an organischem Tensid enthalten.

10. Tablette nach irgendeinem der Ansprüche 1 bis 9, in der die wasserlösliche Substanz, die zusätzlich zu den Teilchen anwesend ist, 25 bis 35% (bezüglich Gewicht der Tablette) an Material mit einer Wasserlöslichkeit von mindestens 50 g/Liter bei 20°C oder Natriumtripolyphosphat, das mehr als 50% seines eigenen Gewichts der wasserfreien Phase I-Form enthält, oder eine Mischung derselben umfasst.

11. Tablette nach Anspruch 10, in der die wasserlösliche Substanz, die zusätzlich zu den Teilchen anwesend ist, 15 bis 35% (bezogen auf Gewicht der Tablette) an Natriumtripolyphosphat umfasst, das mehr als 50% seines eigenen Gewichts der wasserfreien Phase I-Form enthält und partiell hydratisiert ist, so dass es 1 bis 4 Gewichts-% an Hydratationswasser enthält.

12. Tablette nach irgendeinem der Ansprüche 1 bis 11, in der das anionische Tensid in der Hauptsache Natriumalkylbenzolsulfonat ist, gegebenenfalls begleitet von einer geringeren Menge an anderem anionischem Tensid.

13. Tablette nach irgendeinem der Ansprüche 1 bis 12, in der das nichtionische Tensid in der Hauptsache ethoxylierter Fettalkohol ist, gegebenenfalls begleitet von einer geringeren Menge an anderem nichtionischem Tensid.

14. Tablette nach irgendeinem der Ansprüche 1 bis 13, die durch Komprimieren einer einzigen teilchenförmigen Zusammensetzung gebildet ist.

15. Verfahren zur Herstellung einer Detergens-Tablette nach irgendeinem der Ansprüche 1 bis 14, welches umfasst, dass man a) 30 bis 65 Gewichts-% Teilchen, die 25 bis 80 Gewichts-% (dieser Teilchen) an wasserlöslichem Waschkraft-Builder und 20 bis 50 Gewichts-% (dieser Teilchen) an organischem Nicht-Seifen-Tensid, das anionisch ist, und nichtionischen Tensiden in einem Gewichtsverhältnis von 5:1 bis 1,5:1 enthalten, mit b) 15 oder mehr Gewichts-% Material mischt, das von Seife oder organischem Tensid verschieden ist und das eine Löslichkeit in Wasser von mindestens 10g/Liter bei 20°C aufweist, und die Mischung zu einer Tablette komprimiert.

Revendications

1. Pastille détergente de composition particulaire comprimée, dans laquelle la pastille est homogène et comprend un tensioactif organique et un adjuvant de détergence soluble dans l'eau, **caractérisée en ce que** la pastille contient 30 à 65% en poids de particules qui contiennent 25 à 80% en poids (de ces particules) d'un adjuvant de détergence soluble dans l'eau et 20 à 50% en poids (de ces particules) d'un tensioactif organique non savonneux qui est des tensioactifs anioniques et non ioniques selon un rapport massique de 5:1 à 1,5:1, et **en ce que**, en plus desdites particules, la pastille contient 15% en poids (de la pastille) ou plus d'une matière qui est autre qu'un savon ou un tensioactif organique et qui possède une solubilité dans l'eau d'au moins 10 mg/litre à 20°C.

2. Pastille selon la revendication 1, dans laquelle la pastille contient lesdites particules dans une quantité de l'ordre de 41 à 56% en poids.

3. Pastille selon les revendications 1 à 2, dans laquelle le rapport massique entre les tensioactifs anioniques et le tensioactif non ionique est de l'ordre de 1,7:1 à 5:1.

4. Pastille selon l'une quelconque des revendications 1 à 3, dans laquelle le rapport massique entre le tensioactif non ionique et le savon dans lesdites particules est de l'ordre de 10:1 à 30:1.

5. Pastille selon l'une quelconque des revendications 1 à 4, dans laquelle la quantité de tensioactif organique non savonneux dans lesdites particules est de l'ordre de 22 à 45% en poids des particules.

6. Pastille selon l'une quelconque des revendications 1 à 5, contenant du savon dans lesdites particules dans une quantité de 0,2 à 2% en poids des particules.

7. Pastille selon l'une quelconque des revendications 1 à 6, dans laquelle lesdites particules contiennent 30 à 80% en poids (des particules) d'un adjuvant de détergence soluble dans l'eau qui est un sel métallique alcalin d'un phosphate condensé.

8. Pastille selon la revendication 7, dans laquelle le sel d'un phosphate condensé est du tripolyphosphate de sodium.

9. Pastille selon l'une quelconque des revendications 1 à 8, dans laquelle la matière soluble dans l'eau présente en plus desdites particules comprend 15% (en poids de la pastille) ou plus sous la forme d'autres particules qui contiennent moins de 5% de leur propre poids en tensioactif organique.

10. Pastille selon l'une quelconque des revendications 1 à 9, dans laquelle la substance soluble dans l'eau présente en plus desdites particules comprend 25 à 35% (en poids de la pastille) d'une matière possédant une solubilité dans l'eau d'au moins 50 mg/litre à 20°C, ou de tripolyphosphate de sodium contenant plus de 50% de son propre poids sous la forme anhydre de phase I, ou un mélange de ceux-ci.

11. Pastille selon la revendication 10, dans laquelle la substance soluble dans l'eau présente en plus desdites particules comprend 15 à 35% (en poids de la pastille) de tripolyphosphate de sodium contenant plus de 50% de son propre poids sous la forme anhydre de phase I, qui est partiellement hydraté de manière à contenir 1 à 4% en poids d'eau d'hydratation.

12. Pastille selon l'une quelconque des revendications 1 à 11, dans laquelle ledit tensioactif anionique est en majorité du sulfonate de benzène alkyle de sodium, optionnellement accompagné d'une plus petite quantité d'un autre tensioactif anionique.

13. Pastille selon l'une quelconque des revendications 1 à 12, dans laquelle ledit tensioactif non ionique est en majorité un alcool gras éthoxylé, optionnellement accompagné d'une plus petite quantité d'un autre tensioactif non ionique.

14. Pastille selon l'une quelconque des revendications 1 à 13, qui est formée par compression d'une seule composition particulaire.

15. Procédé de fabrication d'une pastille détergente selon l'une quelconque des revendications 1 à 14, qui comprend le mélange

a) de 30 à 65% en poids de particules qui contiennent 25 à 80% en poids (de ces particules) d'un adjuvant de détergence soluble dans l'eau, et 20 à 50% en poids (de ces particules) d'un tensioactif organique non savonneux qui est des tensioactifs anioniques et non ioniques selon un rapport massique de 5:1 à 1,5:1 avec
b) 15% en poids ou plus d'une matière qui est autre qu'un savon ou un tensioactif organique et qui possède une solubilité dans l'eau d'au moins 10 mg/litre à 20°C, et en compactant le mélange en une pastille.

Fig.1a.

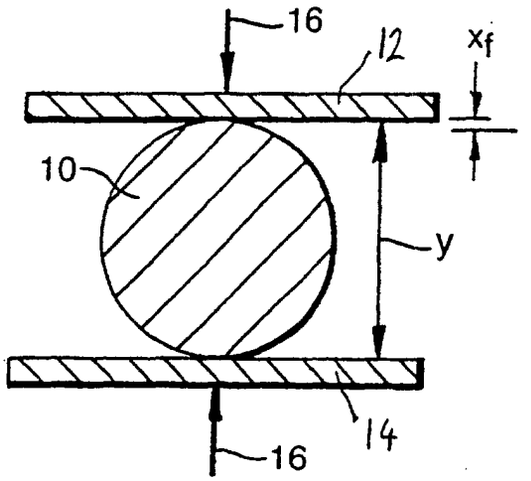


Fig.1b.

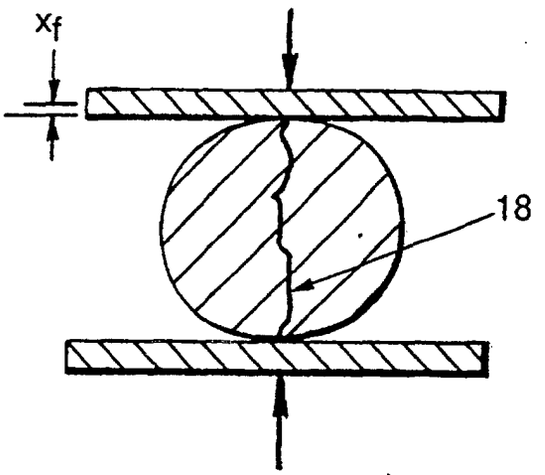


Fig.2.