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(54) Detergent compositions

(57) A tablet of compressed detergent powder contains the inorganic persalt bleach, sodium perborate, in conjunction with one or more bleach-sensitive ingredients - tetraacetylethylenediamine or similar bleach activator, enzyme, fluorescer, or any combination of these - as well as detergent-active compounds, detergency builders and optionally other ingredients. The persalt is not segregated from the bleach-sensitive ingredients but, surprisingly, the tablet is stable with no more loss of bleach, enzyme or fluorescer performance on storage than in a powder of the same composition.

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Description

TECHNICAL FIELD OF INVENTION

[0001] The present invention relates to novel laundry detergent compositions in the form of tablets containing inorganic persalt bleach (sodium perborate) and bleach-sensitive ingredients together without segregation.

BACKGROUND AND PRIOR ART

[0002] Modern high-performance detergent powders for use in automatic washing machines normally contain inorganic persalt bleach as well as certain ingredients, notably bleach activators, enzymes and optical brighteners, that are sensitive to bleach.

[0003] Detergent compositions in tablet form are known in the art, as discussed below, and some products are now on the market. Tablets have several advantages over powdered products: they do not require measuring and are thus easier to handle and dispense into the washload, they are non-dusting and thus their components are less irritating to sensitive skin, and they are more compact, hence facilitating more economical storage.

[0004] However, the ingredients are much more intimately associated with one another in a compressed tablet environment than in a powder, and any adverse interactions and instability will be exacerbated. Hitherto it has not been considered possible to prepare a satisfactory tablet containing both bleach and bleach-sensitive ingredients without some kind of segregation or separation.

[0005] Detergent tablets containing persalt bleach without bleach sensitive ingredients are described, for example, in US 3 953 350 (Kao), JP 60 015 500A (Lion), JP 60 135 497A (Lion) and JP 60 135 498A (Lion). Detergent tablets containing enzyme but no bleach are sold commercially in Spain. GB 911 204 (Unilever) discloses layered detergent tablets containing sodium perborate and certain bleach activators, for example, sodium acetoxybenzene sulphonate and phthalic anhydride, and shows by means of comparative examples that tablets containing those ingredients together, without segregation into separate layers, are unstable.

[0006] Surprisingly, it has now been found that if the bleach activator is a N-diacylated or N,N'-polyacylated amine, notably tetraacetylethylenediamine (TAED), such segregation is unnecessary. Similarly, other bleach-sensitive ingredients, notably enzymes and fluorescers, can also be included without the need for segregation, thus greatly simplifying tablet manufacture. Fully-formulated detergent tablets in accordance with the invention may be made simply by compressing a powder.

DEFINITION OF THE INVENTION

[0007] The present invention accordingly provides a tablet of compressed detergent powder comprising:

(a) sodium perborate;

(b) one or more ingredients sensitive to inorganic persalt bleach selected from

(b1) N-diacylated and N,N'-polyacylated amine bleach activators,
(b2) enzymes, and
(b3) fluorescers;

(c) one or more detergent-active compounds, one or more detergency builders, and optionally other detergent ingredients;
wherein ingredients (a) and (b) are not segregated from one another.

[0008] The invention also provides a process for the preparation of a detergent tablet, which comprises the following steps:

(i) preparing a detergent powder comprising:

(a) sodium perborate;

(b) one or more ingredients sensitive to inorganic persalt bleach selected from

(b1) N-diacylated and N,N'-polyacylated amine bleach activators,

(b2) enzymes, and

(b3) fluorescers;

(c) one or more detergent-active compounds, one or more detergency builders, and optionally other detergent ingredients;

(ii) compressing the powder to form a tablet in which ingredients (a) and (b) are not segregated from one another.

DESCRIPTION OF THE INVENTION

[0009] The detergent tablet of the invention contains as essential ingredients inorganic persalt bleach (sodium perborate), and at least one bleach-sensitive ingredient - acylated amine bleach activator, enzyme, fluorescer - as well as surfactant, detergency builder and optionally other conventional ingredients. The various ingredients are described in more detail below.

[0010] It is an essential feature of the invention that the sodium perborate (a) and the bleach-sensitive ingredient or ingredients (b) are not segregated from one another in the tablet. This means in effect that those ingredients are randomly mixed with one another rather than isolated from one another in specific areas of the tablet, for example, in separate layers or inserts. It does not, however, preclude the presentation of those ingredients in any of the forms commonly used in detergent powders, for example, bleach activators as granules, enzymes as granules or marumes or coated marumes, even though these forms may involve some measure of protection, coating or dilution. However, in the tablet of the present invention, no additional measures are taken, over and above those normal in detergent powders, to keep the sodium perborate (a) and the bleach-sensitive ingredient or ingredients (b) apart.

[0011] According to one preferred embodiment of the invention, the tablet of the invention is substantially homogeneous, that is to say, it is preparable by compression of a single, homogeneously mixed powder.

[0012] It is also within the scope of the invention, however, for the tablet to include one or more separate layers, inserts or other measures in order to segregate ingredients other than (a) and (b). In that case, the ingredients (a) and (b) are randomly mixed and distributed in at least one region of the tablet.

The sodium perborate (a)

[0013] The inorganic persalt bleach employed in the detergent tablet of the invention is sodium perborate. This may be in the form of the monohydrate, the tetrahydrate, a mixture of the two, a partially dehydrated tetrahydrate, or any other stable commercially available form. Sodium perborate monohydrate may be either in high-surface-area or low-surface-area form. The sodium perborate is preferably present in an amount within the range of from 1 to 30 wt%, more preferably from 5 to 20 wt%.

The bleach-sensitive ingredient (b)

[0014] The tablet of the invention contains as an essential ingredient at least one bleach sensitive ingredient selected from one cf the classes (b1), (b2) and (b3). Any combination of more than one bleach-sensitive ingredients from any one or more of these classes may be present.

The bleach activator (b1)

[0015] An activator for an inorganic persalt is an organic compound having one or more reactive acyl residues which cause the formation of peracids, the latter providing a more effective bleaching action at a low wash temperature, eg from 20 to 60°C, than is possible using the inorganic persalt alone.

[0016] The bleach activator used in the tablet of the present invention is an N-diacylated or N,N'-polyacylated amine. These materials are disclosed in GB 855 355 and GB 907 356 (Unilever). In polyamines, at least one nitrogen atom must carry two acyl groups. The preferred bleach activator for use in the tablets of the present invention is tetraacetylethylenediamine (TAED).

[0017] The ratio by weight of the inorganic persalt (sodium perborate) to the bleach activator in the tablet of the invention may vary from about 30:1 to about 1:1, and is preferably from 15:1 to 2:1.

[0018] TAED is conveniently employed in granular form, on an inorganic carrier. The amount of TAED itself in the tablet of the invention is preferably from 1 to 15 wt%, more preferably from 2 to 10 wt%.

The enzyme (b2)

[0019] Various detergency enzymes well-known in the art for their ability to degrade and aid in the removal of various soils and stains may optionally be employed in the detergent tablets of the invention.

[0020] 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. The present invention is particularly concerned with proteolytic enzymes (proteases).

[0021] Preferred proteases are normally solid, catalytically active protein materials which degrade or alter protein types of stains when present as in fabric stains in a hydrolysis reaction. They may be of any suitable origin, such as vegetable, animal, bacterial or yeast origin.

[0022] Proteases of various qualities and origins and having activity in various pH ranges of from 4-12 are available and can be used in the detergent tablet of the invention. Examples of suitable proteases are the subtilisins, which are obtained from particular strains of B. subtilis and B. licheniformis, such as the commercially available subtilisins Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark.

[0023] Particularly suitable for use in the present invention is a protease obtained from a strain of Bacillus having maximum activity throughout the pH range of 8-12, being commercially available, e.g. from Novo Industri A/S under the registered trade-names Esperase (Trade Mark) and Savinase (Trade-Mark). The preparation of these and analogous enzymes is described in GB 1 243 785. Other commercial proteases are Kazusase (Trade Mark) (obtainable from Showa-Denko of Japan), Optimase (Trade Mark) (from Miles Kali-Chemie, Hannover, West Germany), and Superase (Trade Mark) (obtainable from Pfizer of U.S.A.).

[0024] Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in amounts of from about 0.1% to about 3.0% by weight.

The fluorescer (b3)

[0025] Any fluorescer (optical brightener) suitable for use in a detergent powder may be used in the detergent tablet of the present invention.

[0026] The most commonly used fluorescers are those belonging to the classes of diaminostilbene-sulphonic acid derivatives, diarylpyrazoline derivatives and bisphenyl-distyryl derivatives.

[0027] Examples of the diaminostilbene-sulphonic acid derivative type of fluorescer include disodium 4,4'-bis-(2-diethanolamino-4-anilino-s-triazin-6-ylamino) stilbene- 2:2'-disulphonate, disodium 4,4'-bis-(2-morpholino-4-anilino-s-triazin-6-ylamino-stilbene-2:2'-disulphonate, disodium 4,4'-bis-(2,4-dianilino-s-triazin-6-ylamino) stilbene-2:2'-disulphonate, disodium 4,4'-bis-(2-anilino-4-(N-methyl-N-2-hydroxyethylamino)-s-triazin-6-ylamino) stilbene-2,2'-disulphonate, disodium 4,4'-bis-(4-phenyl-2,1,3-triazol-2-yl) stilbene-2,2'-disulphonate, disodium 4,4'-bis(2-anilino-4-(1-methyl-2-hydroxyethylamino)-s-triazin-6-ylamino)stilbene-2,2'-disulphonate and sodium 2-(stilbyl-4''-naptho-1',2':4,5)-1,2,3-triazole-2''-sulphonate.

[0028] Preferred fluorescers are Tinopal (Trade Mark) DMS and 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.

[0029] Other fluorescers suitable for use in the invention include the 1,3-diaryl pyrazolines and the 7-alkylaminocoumarins.

[0030] Fluorescer is preferably present in an amount within the range of from 0.02 to 0.8 wt%, more preferably from 0.03 to 0.5 wt%.

The surfactant system

[0031] The total amount of detergent-active material (surfactant) in the detergent tablet of the invention is suitably from 2 to 50% by weight, and is preferably from 5 to 40% by weight.

[0032] The detergent tablet of the invention may contain one or more soap or non-soap anionic, nonionic, cationic, amphoteric or zwitterionic surfactants, or combinations of these. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

[0033] The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic surfactants and nonionic surfactants.

[0034] Synthetic anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; primary and secondary alkyl sulphates, particularly sodium C_{12}-C_{15} primary alcohol sulphates; olefin sulphonates; alkane sulphonates;

dialkyl sulphosuccinates; and fatty acid ester sulphonates.

[0035] Suitable nonionic detergent 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 either alone or with propylene oxide.

[0036] Specific nonionic detergent compounds are alkyl (C_{6-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols wih ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ehylenediamine. Other so-called nonionic detergent compounds include long-chain tertiary amine oxides, tertiary phosphine oxides, and dialkyl sulphoxides.

[0037] Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{12-15} primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

[0038] 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.

[0039] Detergent compositions suitable for use in automatic fabric washing machines generally contain anionic surfactant, or nonionic surfactant, or a combination of the two in any proportions. Soap may also be present if desired, in any amount from 0 to 40% by weight of the composition. Generally, compositions of this type may successfully be presented in the form of tablets in accordance with the present invention.

The builder system

[0040] The detergent tablets of the invention contain one or more detergency builders, suitably in an amount of from 5 to 80 wt%, preferably from 20 to 80 wt%.

[0041] Especially preferred are alkali metal phosphates, alkali metal aluminosilicates, and combinations thereof.

[0042] Preferred alkali metal phosphates are sodium orthophosphate, sodium pyrophosphate and sodium tripolyphosphate. Sodium tripolyphosphate is especially preferred. It may generally be present in amounts up to 50% by weight of the composition.

[0043] Alkali metal (preferably sodium) aluminosilicates may generally be incorporated in amounts up to 60% by weight of the composition, and may be either crystalline or amorphous or mixtures thereof, having the general formula:

$$0.8\text{-}1.5\ Na_20.\ Al_20_3.0.8\text{-}6\ Si0_2$$

[0044] These materials contain some bound water 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.

[0045] Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in GB 1 429 143 (Proctor & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof. Especially preferred is zeolite 4A.

[0046] Other builders may also be included in the detergent tablet of the invention if necessary or desired: suitable organic or inorganic water-soluble or water-insoluble builders will readily suggest themselves to the skilled detergent formulator. Inorganic builders that may be present include alkali metal (generally sodium) carbonate; while organic builders include nitrilotriacetates, citrates and carboxymethyloxysuccinates.

[0047] Especially preferred supplementary builders are polycarboxylate polymers, especially those containing (meth)acrylate and/or maleate units. These polymers also provide certain additional benefits, for example, reduced soil redisposition. The polymers are suitably used in amounts of from 0.5 to 10% wt%, more preferably from 1 to 6 wt%. The polymers may be in acid form or in wholly or partially neutralised salt form. Preferred polymers are homopolymers and copolymers of acrylic acid. Of especial interest are polyacrylates, acrylic/maleic acid copolymers, and acrylic phosphinates: acrylic/maleic copolymers, for example, Sokalan (Trade Mark) CP5 ex BASF are especially preferred.

Other ingredients

[0048] Further ingredients which can optionally be employed in the detergent tablet of the invention include anti-redeposition agents such as sodium carboxymethylcellulose, polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose; stabilisers such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate; fabric-softening agents; lather control agents or lather boosters as appropriate; heavy metal sequestrants such as EDTA; perfumes; colourants; and inorganic salts such as sodium and magnesium sulphate. Sodium sulphate may if desired be present as a filler material in amounts up to 40% by weight of

the composition; however as little as 10% or less by weight of the composition of sodium sulphate, or even none at all, may be present.

[0049] An antifoam material is advantageously included in the detergent tablet of the invention, especially if the 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). 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 absorbent water-soluble carbonate-based inorganic carrier material. Antifoam granules may be present in any amount up to 5% by weight of the composition.

[0050] It may also be desirable to include in the detergent tablet of the invention an amount of an alkali metal silicate, particularly sodium ortho-, meta- or preferably neutral or alkaline silicate. The presence of such alkali metal silicates at levels, for example, of 0.1 to 10 wt%, may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some measure of building and giving processing benefits.

Preparation of the detergent tablet

[0051] The detergent compositions of the invention are made into tablet form by compression of a powder. As previously mentioned, according to one preferred embodiment of the invention the tablets of the invention are substantially homogeneous, that is to say, they are formed by compression of a single, substantially homogeneous powder without the addition of inserts, separate layers containing segregated ingredients, coatings, etc.; this greatly facilitates manufacture. However, it is also within the scope of the invention, for example, for the starting powder to contain inserts or other local regions of segregation that will persist into the tablet; or for two or more different powders to be compressed to form separate layers in the final tablet; the only proviso being that the ingredients (a) and (b) should not be segregated.

[0052] The powder itself may be made by any suitable method, for example, mixing, granulation, or any combination of these, optionally with spray-on of liquid ingredients. For example, solid ingredients may be dry-mixed, water and/or liquid ingredients added, the mixture granulated, and then dried. Another method involves spray-drying an aqueous slurry of detergent components to form granules, and subsequently post-dosing heat-sensitive ingredients, such as bleach and enzymes; again liquid ingredients may be sprayed on at any suitable stage in the process.

[0053] One preferred process involving spray-drying comprises the steps of:

(i) spray-drying an aqueous slurry to form a base powder including one or more detergent-active compounds and one or more detergency builders, any fluorescer (b3) to be included, and other ingredients as desired;

(ii) admixing to the base powder the sodium perborate (a), any N-diacylated or N,N'-polyacylated amine bleach activator (b1) and/or enzyme (b2) to be included, and optionally other ingredients as desired, to form a homogeneous powder;

(iii) compressing the powder to form a tablet.

Dosage forms

[0054] The detergent tablet of the invention may be, and preferably is, formulated for use as a complete heavy-duty fabric washing composition. The consumer does not need to use a mix of tablets having different compositions.

[0055] Although one tablet may contain sufficient of all the components to provide the correct amount required for an average washload, it is preferred that each tablet should contain a submultiple quantity of the composition required for average washing conditions, so that the consumer may vary the dosage according to the size and nature of the washload. For example, tablet sizes may be chosen such that two tablets are sufficient for an average washload; one or more further tablets may be added if the washload is particularly large or soiled; and one only tablet may be used if the load is small or only lightly soiled.

EXAMPLES

[0056] The invention will now be illustrated by the following non-limiting Examples in which parts are percentages are by weight. Examples and compositions identified by numbers are within the invention, while those identified by letters are comparative.

Example 1

[0057] A zero-phosphate detergent base powder containing fluorescer was prepared to the following composition by spray-drying, followed by spray-on of the 3EO nonionic surfactant:

	%
Linear alkylbenzene sulphonate	9.2
C_{12}-C_{15} 7EO nonionic surfactant	4.2
C_{13}-C_{15} 3EO nonionic surfactant	9.2
Fatty acid soap	2.5
Zeolite 4A (anhydrous basis)	36.8
Acrylic/maleic copolymer (Sokalan* CP5)	6.0
Sodium silicate	0.7
Sodium carboxymethyl cellulose	0.8
Fluorescers (Tinopal** DMS and Tinopal** CBS-X)	0.3
Sodium carbonate	12.9
Water	to 100.0

*Trade Mark; ex BASF AG, Ludwigshafen, Germany.
**Trade Mark; ex Ciba-Geigy AG, Basel, Switzerland.

[0058] Tablets and powders were prepared to the compositions given below. The enzyme was the granular protease Savinase (Trade Mark) 6.0T, the sodium perborate was high-surface-area (5.9 m^2/g) monohydrate, and the TAED was in granular form (83 wt% TAED, 17 wt% inert zero-phosphate carrier).

Example	1 Tablet	P Powder
Base	85.4	85.4
Enzyme	1.0	1.0
Perborate	10.0	10.0
TAED granules	3.6	3.6

[0059] Tablets were made by compressing the powders at 1.5 kN by means of an Instron (Trade Mark) constant speed extensiometer, using a crosshead speed of 100 mm/min. Each tablet weighed 60 g and had a diameter of 53 mm and a thickness of about 25 mm.

[0060] The bleach, fluorescer and enzyme performances of the tablets and powders were compared for the freshly made compositions, and after 1 week's and 2 weeks' storage under severe conditions (37°C and 70% relative humidity, unprotected, no carton). The comparisons were carried out by washing selected testcloths in the tergotometer using a 40°C isothermal wash for 15 minutes in 1 litre of 12° (French) hard water. The tablets were crushed prior to the wash, and dosed at 5 g/litre; the powders were also dosed at 5 g/litre.

[0061] Table 1 shows the enzyme results, obtained using an enzyme-sensitive testcloth (casein soil on polyester/cotton fabric), expressed as reflectance differences (delta R at 460 nm).

Table 1

Example	Time (weeks)		
	0	1	2
1 (tablet)	26.9	26.8	26.9
P (powder)	27.0	26.6	26.4

[0062] There was no evidence of increased enzyme decomposition in the tablet as compared with the powder, despite the close proximity to the bleach ingredients.

[0063] Table 2 shows the fluorescer results, obtained using a fluorescer-sensitive testcloth, and expressed as the difference between the reflectances (delta R 460) in the presence and in the absence of ultraviolet light.

Table 2

Example	Time (weeks)		
	0	1	2
1 (tablet)	15.7	16.6	15.7
P (powder)	15.6	16.2	15.3

[0064] Again, no significant differences between tablet and powder were found.

[0065] Table 3 shows the bleach results, obtained using a tea-stained cotton testcloth and expressed as reflectance differences (delta R at 460 nm).

Table 3

Example	Time (weeks)		
	0	1	2
1 (tablet)	5.3	4.3	4.0
P (powder)	3.8	3.1	3.2

[0066] Surprisingly, the results were consistently better for the tablet than for the powder of identical composition.

Example 2

[0067] A phosphate-built base detergent base powder containing fluorescer was prepared to the following composition by spray-drying, followed by spray-on of the 3EO nonionic surfactant:

	%
Linear alkylbenzene sulphonate	8.3
C_{12}-C_{15} 7EO nonionic surfactant	5.3
C_{13}-C_{15} 3EO nonionic surfactant	3.5
Fatty acid soap	0.7
Sodium tripolyphosphate	29.3
Acrylic/maleic copolymer (Sokalan CP5)	1.2
Sodium silicate	7.0

(continued)

	%
Sodium carboxymethyl cellulose	0.4
EDTA	0.15
Fluorescers (Tinopal DMS and Tinopal CBS-X)	0.35
Sodium carbonate	5.9
Sodium sulphate, water	to 100.0

[0068] Tablets and powders were prepared as described in Example 1 to the compositions given below. The enzyme, sodium perborate monohydrate and TAED were as in Example 1.

Example	2 Tablet	Q Powder
Base	85.4	85.4
Enzyme	1.0	1.0
Perborate	10.0	10.0
TAED granules	3.6	3.6

[0069] The bleach, fluorescer and enzyme performances of the tablets and powders were compared as described in Example 1. Enzyme results are shown in Table 4, fluorescer results in Table 5, and bleach results in Table 6.

Table 4

(enzyme)			
Example	Time (weeks)		
	0	1	2
2 (tablet)	26.6	27.1	25.8
Q (powder)	26.8	26.1	25.7

Table 5

(fluorescer)			
Example	Time (weeks)		
	0	1	2
2 (tablet)	16.1	15.4	15.6
Q (powder)	15.0	15.5	15.3

[0070] Again, no significant differences between tablet and powder were found.

Table 6

(bleach)			
Example	Time (weeks)		
	0	1	2
2 (tablet)	6.0	5.4	5.0
Q (powder)	5.6	5.2	4.3

[0071] Bleach loss on storage was observed, as expected, but surprisingly the results were consistently better for the tablet than for the powder.

Examples 3 to 5

[0072] A further series of comparisons was carried out using the zero-phosphate base powder of Example 1, and three different types of sodium perborate:
high-surface-area (5.9 m^2/g) monohydrate,
low-surface-area (3.2 m^2/g) monohydrate, and
tetrahydrate.

[0073] Tablets and powders were prepared to the following compositions, Examples 3, 4 and 5 being tablets and Examples R, S and T being powders:

Example	3, R	4, S	5, T
Base	89.0	89.0	84.0
Enzyme	1.0	1.0	1.0
Perborate :			
Mono (HSA)	10.0	-	-
Mono (LSA)	-	10.0	-
Tetra	-	-	15.0

[0074] Bleach and enzyme performances of the fresh compositions, and after storage for 2 and 4 weeks (37°C, 70% relative humidity), were compared by the methods described in Example 1. The results are shown below in Tables 7 (bleach) and 8 (enzyme).

Table 7

(bleach)		
Example	Time (weeks)	
	0	4
3 (tablet)	2.3	0.9
R (powder)	1.5	1.1
4 (tablet)	2.0	0.8
S (powder)	2.4	0.5
5 (tablet)	1.3	1.6
T (powder)	2.3	0.8

[0075] When perborate monohydrate was present, bleach performance always deteriorated after 4 weeks' storage, as expected, but that was no worse in the tablets than in the powders, and, surprisingly, there was less loss of performance from the tablet than from the powder in the case of the low-surface-area material. When the perborate was in tetrahydrate form, an even better result for the tablet was obtained.

Table 8

(enzyme)			
Example	Time (weeks)		
	0	2	4
3 (tablet)	29.1	31.8	34.1
R (powder)	33.6	34.5	32.8
4 (tablet)	33.4	32.3	33.2
S (powder)	34.3	34.1	34.2
5 (tablet)	33.0	32.2	33.8
T (powder)	33.4	33.1	32.9

[0076] No significant differences were observed.

Examples 6 to 8

[0077] The exercise of Examples 3 to 5 was repeated using a similar series of compositions additionally containing TAED (83 wt% zero-phosphate granules as used in Example 1). Tablets and powders were prepared to the following compositions, Examples 6, 7 and 8 being tablets and Examples U, V and W being powders:

Example	6, U	7, V	8, W
Base	85.4	85.4	80.4
Enzyme	1.0	1.0	1.0
Perborate :			
Mono (HSA)	10.0	-	-
Mono (LSA)		10.0	-
Tetra		-	15.0
TAED granules	3.6	3.6	3.6

[0078] Bleach and enzyme performances of the fresh compositions, and after storage for 2 and 4 weeks (37°C, 70% relative humidity), were compared as described in Example 1, the results being shown below in Tables 9 (bleach) and 10 (enzyme).

Table 9

(bleach)		
Example	Time (weeks)	
	0	4
6 (tablet)	2.7	3.3
U (powder)	4.4	3.0

Table 9 (continued)

(bleach)		
Example	Time (weeks)	
	0	4
7 (tablet)	3.8	3.2
V (powder)	4.0	3.5
8 (tablet)	4.0	3.3
W (powder)	4.1	3.6

[0079] No significant differences between tablets and powders were observed.

Table 10

(enzyme)			
Example	Time (weeks)		
	0	2	4
6 (tablet)	33.4	31.7	34.1
U (powder)	32.6	33.8	32.4
7 (tablet)	33.5	32.5	33.8
V (powder)	32.2	32.7	33.5
8 (tablet)	33.3	33.6	33.6
W (powder)	33.3	33.9	32.6

[0080] No significant differences were observed.

Examples 9 and 10

[0081] Examples 3 and 6 were repeated using the phosphate-built base powder of Example 2 instead of the zero-phosphate base powder of Example 1. Tablets and powders were prepared to the following compositions, Examples 9 and 10 being tablets and Examples X and Y being powders:

Example	9, X	10, Y
Base	89.0	85.4
Enzyme	1.0	1.0
Perborate (mono, HSA)	10.0	10.0
TAED granules	-	3.6

[0082] Bleach and enzyme performances of the fresh compositions, and after storage for 2 and 4 weeks (37°C, 70% relative humidity), were compared as described in Example 1, the results being shown below in Tables 11 (bleach) and 12 (enzyme).

Table 11

(bleach)		
Example	Time (weeks)	
	0	4
9 (tablet)	1.7	1.9
X (powder)	2.4	1.1
10 (tablet)	5.7	4.4
Y (tablet)	5.0	4.1

[0083] The tablet showed better bleach performance than the corresponding powder after 4 weeks' storage.

Table 12

(enzyme)			
Example	Time (weeks)		
	0	2	4
9 (tablet)	34.1	32.4	32.0
X (powder)	32.6	33.5	31.4
10 (tablet)	34.3	32.4	31.4
Y (powder)	32.1	31.3	32.8

[0084] No significant differences were observed.

Example 11

[0085] A phosphate-built detergent powder containing sodium perborate tetrahydrate bleach in conjunction with proteolytic enzyme (Savinase) and fluorescers was prepared, by spray-drying and postdosing, to the following composition:

		%
Linear alkylbenzene sulphonate		18.0
C_{12}-C_{15} 7EO nonionic surfactant		8.0
Fatty acid soap		2.0
Sodium tripolyphosphate		30.0
Acrylic/maleic copolymer (Sokalan CP5)		1.5
Alkaline sodium silicate		10.0
Sodium carboxymethyl cellulose		0.5
EDTA		0.25
Fluorescers:	Tinopal DMS	0.13
	Tinopal CBS-X	0.08

(continued)

	%
Sodium sulphate	3.6
Sodium perborate tetrahydrate	10.0
Enzyme (Savinase 6CM)	0.75
Antifoam granules	3.0
Perfume	0.22
Moisture, salts	to 100.0

[0086] The powder had a bulk density of 400-425 g/litre. It was compressed to approximately half its volume, using a pilot-scale tabletting machine, to form tablets weighing 32 g and having a diameter of 50 mm and a height of 23 mm.

[0087] The tablets displayed effective washing and cleaning performance in the washing machine, two tablets being an appropriate dosage for soft water and three for hard water.

Example 12

[0088] A phosphate-built detergent powder containing sodium perborate tetrahydrate bleach in conjunction with TAED granules, proteolytic enzyme (Savinase) and fluorescers was prepared, by spray-drying and postdosing, to the following composition, and then compressed as described in Example 11 to form tablets:

		%
Linear alkylbenzene sulphonate		18.0
C_{12}-C_{15} 7EO nonionic surfactant		8.0
Fatty acid soap		2.0
Sodium tripolyphosphate		30.0
Acrylic/maleic copolymer (Sokalan CP5)		1.5
Alkaline sodium silicate		10.0
Sodium carboxymethyl cellulose		0.5
EDTA		0.25
Fluorescers:	Tinopal DMS	0.13
	Tinopal CBS-X	0.08
Sodium sulphate		1.3
Sodium perborate tetrahydrate		10.0
TAED granules (83 wt% active)		2.4
Enzyme (Savinase 6CM)		0.75
Antifoam granules		3.0
Perfume		0.22
Moisture, salts		to 100.0

[0089] The tablets displayed effective cleaning and washing performance when used at a dosage of two to three tablets per wash.

Example 13

[0090] A phosphate-built detergent powder with enhanced bleaching performance, containing sodium perborate monohydrate bleach in conjunction with TAED, proteolytic enzyme (Savinase) and fluorescers, was prepared to the following composition by spray-drying and postdosing:

		%
Linear alkylbenzene sulphonate		18.0
C_{12}-C_{15} 7EO nonionic surfactant		8.0
Fatty acid soap		2.0
Sodium tripolyphosphate		30.0
Acrylic/maleic copolymer (Sokalan* CP5)		1.5
Alkaline sodium silicate		10.0
Sodium carboxymethyl cellulose		0.5
EDTA		0.25
Fluorescers:	Tinopal DMS	0.13
	Tinopal CBS-X	0.08
Salts		4.24
Water		8.0
Sodium perborate monohydrate		10.0
TAED		3.33
Enzyme (Savinase 6CM)		0.75
Antifoam granules		3.0
Perfume		0.22

*Trade Mark; ex BASF AG, Ludwigshafen, Germany.

[0091] The powder was compressed as described in Example 11 to form tablets, each weighing 26-28 g, having a diameter of 40 mm and a thickness of 26 mm. The tablets displayed effective washing and cleaning performance when used at a dosage of two or three tablets per wash.

Examples 14 and 15

[0092] The following zero-phosphate detergent powders, containing sodium perborate monohydrate bleach in conjunction with TAED, proteolytic enzyme (Savinase) and fluorescers, prepared by spray-drying and postdosing, are suitable for compression to form 40 g tablets according to the present invention.

	14	15
Linear alkylbenzene sulphonate	16.0	6.0
C_{12}-C_{15} 7EO nonionic surfactant	3.0	4.5
C_{13}-C_{15} 3EO nonionic surfactant	5.0	4.0
Fatty acid soap	2.0	-
Zeolite 4A (anhydrous basis)	30.0	37.0

(continued)

		14	15
Acrylic/maleic copolymer (Sokalan CP5)		4.0	5.0
Sodium carboxymethyl cellulose		0.5	0.5
Fluorescers:	Tinopal DMS-X	0.13	0.13
	Tinopal CBS-X	0.08	0.07
Sodium carbonate		9.0	14.9
Sodium perborate monohydrate		10.0	7.5
TAED granules (83%)		4.5	4.5
Enzyme (Savinase 6.0T)		1.0	1.0
Antifoam granules		3.0	1.5
Perfume		0.4	0.4
Water and minor ingredients to 100.0			

Claims

1. A tablet of compressed detergent powder characterised in that it comprises:

(a) sodium perborate

(b) one or more ingredients sensitive to inorganic persalt bleach selected from

(b1) N-diacylated and N,N'-polyacylated amine bleach activators,
(b2) enzymes, and
(b3) fluorescers;

(c) one or more detergent-active compounds, one or more detergency builders, and optionally other detergent ingredients;
wherein ingredients (a) and (b) are not segregated from one another.

2. A detergent tablet in claim 1, wherein the sodium perborate (a) is present as sodium perborate monohydrate, and containing up to 60% by weight of alkali metal aluminosilicate detergency builder.

3. A detergent tablet as claimed in either claim 1 or claim 2, characterised in that the sodium perborate (a) is present in an amount within the range of from 1 to 30 wt%.

4. A detergent tablet as claimed in any preceding claim, comprising a bleach activator (b1), characterised in that the weight ratio of sodium perborate (a) to bleach activator (b1) is within the range of from 30:1 to 1:1.

5. A detergent tablet as claimed in either claim 1 or claim 2, characterised in that it comprises as bleach activator tetraacetylethylenediamine.

6. A detergent tablet as claimed in claim 5, characterised in that the tetraacetylethylenediamine is in granular form and is present in an amount within the range of from 1 to 15 wt%.

7. A detergent tablet as claimed in claim 6, characterised in that the tetraacetylethylenediamine is in granular form and is present in an amount within the range of from 2 to 10 wt%.

8. A detergent tablet as claimed in any preceding claim, characterised in that it comprises an enzyme (b2) in granular form in an amount within the range of from 0.1 to 3.0 wt%.

9. A detergent tablet as claimed in claim 8, characterised in that the enzyme is a protease.

10. A detergent tablet as claimed in any preceding claim, characterised in that it is substantially homogenous.

11. A process for the preparation of a detergent tablet, characterised by the steps of:

(i) preparing a detergent powder comprising:

(a) sodium perborate

(b) one or more ingredients sensitive to inorganic persalt bleach selected from

(b1) N-diacylated and N,N'-polyacylated amine bleach activators,
(b2) enzymes, and
(b3) fluorescers;

(c) one or more detergent-active compounds, one or more detergency builders, and optionally other detergent ingredients;

(ii) compressing the powder to form a tablet in which ingredients (a) and (b) are not segregated from one another.

12. A process as claimed in claim 11, wherein the sodium perborate (a) is present as sodium perborate monohydrate and, wherein the detergent contains up to 60% by weight of alkali metal aluminosilicate detergency builder.

13. A process as claimed in either claim 11 or claim 12, characterised by the steps of:

(i) spray-drying an aqueous slurry to form a base powder including one or more detergent-active compounds and one or more detergency builders, any fluorescer (b3) to be included, and other ingredients as desires;
(ii) admixing to the base powder the sodium perborate (a), any N-diacylated or N,N'-polyacylated amine bleach activator (b1) and/or enzyme (b2) to be included, and optionally other ingredients, as desired, to form a homogeneous powder;
(iii) compressing the powder to form a tablet.