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(54) Title: DETERGENT COMPOSITIONS (57) Abstract A particulate detergent composition or component of high bulk density (at least 600 g/l), comprising at least 10 % by weight of detergent surfactant and from 10 to 70 % by weight of detergency builder, is composed of at least two, and preferably at least three, different granular components: (i) granules comprising at least 60 % by weight of anionic surfactant, (ii) granules comprising at least 20 % by weight of nonionic surfactant, and less than 10 % by weight of aluminosilicate, (iii) optionally, granules comprising detergency builder.		

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DETERGENT COMPOSITIONS

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Technical field

The present invention relates to a medium to high bulk density particulate detergent composition.

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Background

Traditionally, particulate detergent compositions have been manufactured by the spray drying process, in which a slurry of components such as anionic detergent active, builder material and optionally non-anionic detergent active is manufactured and then dried by atomising it and spraying it into a stream of air at high temperature. The spray-dried compositions are found in practice to have bulk densities less than 600 g/l. There are limits on the quantity of anionic detergent active that can be included due to the need to form a slurry before spray-drying. The resultant spray-dried granules may be used directly as a detergent composition or other components may be post-dosed, for example heat or moisture sensitive components, to provide a complete powder composition.

In recent years, a number of detergent powder manufacturing processes have been developed in which a spray-drying tower is not used. Such so called non-tower route (NTR) processes typically involve granulation of anionic detergent active and builder in a high or medium speed mixer/densifier,

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typically in the presence of a liquid binder such as water or non-anionic detergent active. High detergent active compositions having medium to high bulk densities (500-900 g/l) have been produced by such non-tower processes.

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However, it has been found that such so called concentrated products may have unsatisfactory dispensing properties in wash water, particularly in automatic washing machines. Problems have been encountered such as poor dispersion of the powder into the wash water in the dispenser drawer of a washing machine. A gritty, viscous mass may remain in the dispenser drawer. Further, powder compositions entrained in the wash water may not break up and disperse adequately. Undissolved particles of powder compositions may remain in the wash water. These can adhere to clothes and cause local damage. For example, where the detergent composition contains bleach, an undissolved mass of composition can adhere to clothing and, due to the locally high concentration of bleach, damage the clothing. Undissolved powder composition can remain on the clothes after washing.

Summary of the invention

25 It is an object of the present invention to provide a detergent powder composition having a medium to high bulk density (a bulk density of at least 600 g/l) and a moderate to high anionic detergent active content.

30 The present inventors have discovered that the dispersion problems of particulate detergent compositions can be due to unfavourable properties of some of the components, such as anionic surfactant, or due to unfavourable interactions

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between different components which occur in the particles. The inventors have discovered that it is beneficial to concentrate the components having undesirable properties into a smaller number of particles. This entails providing particles of high concentration of the component which has the undesirable property. Further, it is desired to separate components which would show unfavourable interactions if they were included in the same particle.

Accordingly, the present invention provides a particulate detergent composition or component having a bulk density of at least 600 g/l and comprising at least 10% by weight (preferably at least 12% by weight) of organic detergent surfactant and from 10 to 70% by weight (preferably from 15 to 70% by weight) of detergency builder, the detergent composition or component being composed of at least two, and preferably at least three, granular components:

(i) granules comprising at least 60% by weight of anionic surfactant ("anionic surfactant granules"),

(ii) granules comprising at least 20% by weight of nonionic surfactant and less than 10% by weight aluminosilicate ("nonionic surfactant granules"), and

(iii) optionally, granules comprising up to 100% (preferably up to 90%) by weight of detergency builder and optionally from 0 to 10% of nonionic or anionic surfactant ("builder granules").

The anionic surfactant granules (i) are preferably present in an amount of from 1 to 70% by weight.

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The nonionic surfactant granules (ii) are preferably present in an amount of from 1 to 30% by weight of the composition, more preferably from 1 to 50% by weight.

- 5 The optional builder granules are preferably present in an amount of from 5 to 80% by weight, and more preferably in an amount of at least 15% by weight.

10 The composition limits on the individual granules according to the invention have been found to provide detergent compositions having surprisingly reduced problems of residues in the wash.

It has been found that there is a particularly
15 disadvantageous interaction between nonionic surfactant and aluminosilicate builder, which leads to problems of residues in the wash discussed above. The composition of the present invention allows this problem to be overcome. Further,
20 there can be problems due to unfavourable interaction between aluminosilicate builder and anionic surfactant. The effects of concentrating anionic surfactant in the anionic surfactant granule will be discussed further below.

25 The total quantity of detergent surfactant in the compositions of the invention is preferably at least 12% by weight, and the quantity of nonionic surfactant is preferably from 1 to 40% by weight, more preferably from 1 to 30% by weight.

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The anionic surfactant granules (i)

The anionic surfactant granules preferably comprise from 60 to 90% by weight of anionic surfactant.

5

The anionic surfactant granules may also contain nonionic surfactant. The anionic surfactant granules may also contain minor ingredients such as water, sodium carboxymethylcellulose, fluorescers, dyes, etc.

10

The anionic surfactant granules may optionally comprise from 0 to 40% by weight of detergency builder. The builder material may comprise soluble builder such as salts (preferably alkali metal salts, particularly preferably sodium salts) of tripolyphosphate, carbonate, silicate, sesquicarbonate, citrate or mixtures thereof, or burkeite (a double salt of sodium sulphate and sodium carbonate), NTA, polycarboxylic acid monomer, polycarboxylic acid polymer, polycarboxylic acid/maleic acid copolymer or mixtures thereof.

20

The builder may comprise insoluble builder such as aluminosilicate. The aluminosilicate may comprise zeolite, in particular zeolite MAP, zeolite 4A, amorphous aluminosilicate and mixtures thereof. It is particularly preferred, however, that the quantity of aluminosilicate builder is low. Preferably, aluminosilicate builder provides less than 15% by weight of the anionic surfactant particles, more preferably less than 10%.

30

The anionic surfactant granules may be manufactured by any suitable process. Preferably, such granules are manufactured by mixing the components in a high speed mixer

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to agglomerate the components. Suitable mixers will be discussed further below.

Processes for producing granules containing high quantities
5 of anionic surfactant are set out in WO 96/06916A and
WO 96/06917A (Unilever).

The method of WO 97/32002A (Unilever) is particularly
preferred. In this method, a paste material comprising
10 water and an anionic surfactant, or a mixture of acid
surfactant precursor and alkaline neutralising agent, is fed
into a drying zone, the paste material being heated in the
drying zone to reduce the water content thereof and the
paste material being subsequently cooled in a cooling zone
15 to form detergent particles, a layering agent being
introduced into the cooling zone during the cooling step.
Alternatively, a paste material comprising water and an
anionic surfactant, or a mixture of acid surfactant
precursor and alkaline neutralising agent fed into a drying
20 zone, the material being heated in the drying zone to reduce
the water content thereof and the material being
subsequently cooled in a cooling zone to form detergent
particles, the material being treated in the cooling zone
with a stream of cooling gas. This process can provide
25 detergent particles comprising at least 60% by weight of the
particle of an anionic surfactant and not more than 5% by
weight of the particle of water. The particles are coated
with layering agent.

30 The detergent particles may comprise anionic surfactant in
an amount of at least 60% by weight of the particle, the
particles being coated with layering agent and having a
porosity of from 0 to 25% by volume of the particle and a

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particle size distribution such that at least 80% of the particles have a particle size of 180-1500 microns. The layering agent may comprise an aluminosilicate, a silica or a mixture thereof. The layering agent may be dosed into the cooling zone at a weight ratio of from 1:5 to 1:20 relative to the finished particles. The anionic surfactant may be formed in situ by neutralisation of a free acid with neutralising agents such as sodium hydroxide solution or sodium carbonate.

10

The nonionic surfactant granules (ii)

The nonionic surfactant granules comprise at least 20% by weight of nonionic surfactant.

15

The quantity of aluminosilicate builder must be less than 10% by weight. This helps to avoid unfavourable generation of residues and poor dispersing properties in wash water.

20

The nonionic surfactant particles preferably contain less than 10% by weight of anionic surfactant, and preferably substantially no anionic surfactant.

Nonionic surfactant particles for use in the present invention generally fall into one of two classes.

25

The first class comprises nonionic surfactant carried on water-soluble carrier material. Suitable carrier materials include burkeite, sodium sesquicarbonate, sodium carbonate, sodium sulphate and mixtures thereof. A nonionic surfactant granule comprising water-soluble carrier preferably

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comprises from 20 to 50% by weight, preferably from 25 to 40% by weight, of nonionic surfactant.

5 The water-soluble carrier material is preferably present at a level exceeding 40% by weight, preferably 60% by weight or more.

10 The second class of nonionic surfactant granule comprises water-insoluble carrier material. The insoluble carrier material may comprise silica or aluminosilicate, such as zeolite. However, it is essential that the quantity of aluminosilicate is less than 10% by weight. Where an insoluble carrier material is used, the quantity of nonionic surfactant may exceed 55% by weight of the granule.

15 Structuring agents such as polyethylene/poly-propylene glycol of average molecular weight in the region 4000-12000, sodium soap, polyvinyl alcohol of average molecular weight in the range 30 000-200 000, alkaline metal succinate etc. 20 may be present. The preferred quantity of structuring agent is in the region of from 0.5 to 10% by weight.

Nonionic-surfactant-containing granules comprising 55% by weight or more of nonionic surfactant, at least 5% by weight 25 of silica of oil absorption capacity of 1.0 ml/g and less than 10% by weight of aluminosilicate are disclosed in our copending application of even date (reference C3777) entitled "Detergent Compositions Containing Nonionic Surfactant Granule". These granules can be manufactured by 30 mixing together components in a granulator (for example an Eirich RVO2 Granulator). Alternatively, 70 to 100% by weight of the solid components and 70 to 95% by weight of the nonionic surfactant can be mixed together in a first

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step, the remainder of the solid components and nonionic surfactant being added in a second step, preferably under moderate shear. In the second process, the majority of the structurant is preferably added in the second step.

5

As indicated previously, the nonionic surfactant granules are preferably present in an amount of from 1 to 50%, preferably from 1 to 30%, by weight of the composition. They may suitably provide 20% or more of the composition.

10

Optional builder granules (iii)

The optional builder granule may contain soluble builder such as sodium tripolyphosphate, sodium carbonate, sodium silicate, NTA, sodium sesquicarbonate, burkeite, sodium citrate, polycarboxylic acid monomer, polycarboxylic acid polymer/copolymer or mixtures thereof.

20 The optional builder granule may also comprise aluminosilicate, preferably crystalline aluminosilicate such as zeolite. The builder granule is preferably present in an amount of from 5 to 80% by weight, and may suitably represent 15% by weight or more of the composition, more
25 preferably 18% by weight or more.

The builder granule optionally contains additional nonionic and/or anionic surfactant selected from the examples above. The total quantity of surfactant in the builder granule is
30 preferably less than 10% by weight.

The builder granule may also comprise layered silicate, available, for example, as SKS-6 (Hoechst).

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Any suitable means may be used to prepare the builder granules. For example, the builder granules may be manufactured by spray drying a slurry of the components. Alternatively, the components may be placed in a high speed mixer/densifier and granulated in the presence of liquid binder such as water or solution of polymer, such as builder polymer, or solution of salt, such as silicate.

10 Other ingredients

The detergent composition of the present invention may consist only of the anionic granule, the nonionic granule and, optionally, the builder granule.

15

However, other detergent ingredients may be postdosed to the composition to provide detergent benefits, in which case the composition of the invention may be regarded as a "detergent component" rather than a full "detergent composition".

20

Examples of ingredients which may be postdosed are bleach ingredients, bleach precursor, bleach catalyst, bleach stabiliser, photobleaches, alkali metal carbonate, water-soluble crystalline or amorphous alkaline metal silicate, layered silicates, anti-redeposition agents, soil release polymers, dye transfer inhibitors, fluorescers, inorganic salts, foam control agents, foam boosters, proteolytic, lipolytic, amylitic and cellulytic enzymes, dyes, speckles, perfume, fabric conditioning compounds and mixtures thereof.

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Preferably the detergent composition contains 40 to 85% by weight, in total, of the anionic surfactant granules, the

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nonionic surfactant granules and, if present, the builder granules.

5 In each case, there may be more than one type of anionic surfactant granule, nonionic surfactant granule and builder granule.

10 In the present specification, the term "granule" is used to denote a solid particle of size greater than 200 micrometers. Preferably, such granules will be the direct product of a spray drying or agglomeration process.

Preparation of the compositions of the invention

15

The invention further provides a method of manufacturing a detergent powder composition or component as previously defined, comprising the steps of:

20 (i) manufacturing granules comprising at least 60% by weight of anionic surfactant,

(ii) manufacturing granules containing at least 20% by weight nonionic surfactant and less than 10% by weight aluminosilicate,

25

(iii) optionally manufacturing granules comprising up to 100% by weight of builder and optionally from 0 to 10% by weight of nonionic or anionic surfactant, and
30 mixing the granules produced in steps (i) and (ii), and optionally the granules produced in step (iii).

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Detergent Ingredients

The detergent compositions of the invention will contain, as essential ingredients, one or more detergent active
5 compounds (surfactants) which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent active compounds, and mixtures thereof.

10 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.

15 The preferred detergent active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds.

Anionic surfactants are well-known to those skilled in the
20 art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅; primary and secondary alkylsulphates, particularly C₈-C₁₅ primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates;
25 dialkyl sulphosuccinates; and fatty acid estersulphonates. Sodium salts are generally preferred.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C₈-C₂₀
30 aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C₁₀-C₁₅ primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles

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of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglyco-sides, glycerol monoethers, and polyhydroxyamides (glucamide).

5 In the compositions of the invention, the total quantity of detergent surfactant in the composition is at least 10% by weight, preferably at least 12% by weight, more preferably at least 15% by weight. The composition may comprise up to 60% by weight of detergent surfactant, preferably up to 50%
10 by weight.

Preferably, the quantity of anionic surfactant is in the range of from 5 to 50% by weight of the total composition. More preferably, the quantity of anionic surfactant is in
15 the range of from 8 to 35% by weight.

Preferably, the quantity of nonionic surfactant is in the range of from 5 to 20% by weight, more preferably from 5 to 15% by weight.

20

Detergent compositions suitable for use in most automatic fabric washing machines generally contain anionic non-soap surfactant, or nonionic surfactant, or combinations of the two in any ratio, optionally together with soap.

25

The anionic surfactant may be produced by neutralising a liquid acid precursor with alkali, such as sodium hydroxide solution or solid sodium carbonate in situ in the granulation process.

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The liquid acid precursor of an anionic surfactant may be selected from the acid precursors of linear alkyl benzene sulphonate, alpha-olefin sulphonate, internal olefin

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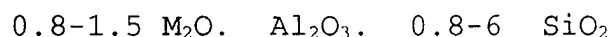
sulphonate, alkyl ether sulphate or fatty acid ether sulphate and combinations thereof.

The anionic surfactants may be primary or secondary alcohol sulphates. Linear or branched primary alcohol sulphates having 10 to 20 carbon atoms are particularly preferred. These surfactants can be obtained by sulphation of the corresponding primary or secondary alcohols, of synthetic or natural origin, followed by neutralisation. Because the acid precursors of alcohol sulphates are chemically unstable, they are not commercially available and they have to be neutralised as quickly as possible after their manufacture.

The compositions of the present invention contain from 10 to 70%, preferably from 15 to 70% by weight, of detergency builder. Preferably, the quantity of builder is in the range of from 15 to 50% by weight.

The detergent composition of the invention may contain a crystalline aluminosilicate, preferably an alkali metal aluminosilicate, more preferably a sodium aluminosilicate.

The aluminosilicate may generally be incorporated in amounts of from 10 to 70% by weight (anhydrous basis), preferably from 25 to 50%. Aluminosilicates are materials having the general formula:



30

where M is a monovalent cation, preferably sodium. These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g.

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The preferred sodium aluminosilicates contain 1.5-3.5 SiO₂ units in the formula above. They can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

5

The zeolite used in the compositions of the present invention may be the commercially available zeolite A (zeolite 4A) now widely used in laundry detergent powders. However, according to a preferred embodiment of the

10 invention, the zeolite incorporated in the compositions of the invention is maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070B (Unilever), and commercially available as Doucil (Trade Mark) A24 from Crosfield Chemicals Ltd, UK.

15

Zeolite MAP is defined as an alkali metal aluminosilicate of zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, preferably within the range of from 0.90 to 1.20.

20 especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00. the calcium binding capacity of zeolite MAP is generally at least 150 mg CaO per g of anhydrous material.

25 The detergent composition may contain crystalline or amorphous water-soluble alkali metal silicate, preferably sodium silicate having a SiO₂:Na₂O mole ratio within the range of from 1.6:1 to 4:1, 2:1 to 3.3:1.

30 The water-soluble silicate may be present in an amount of from 1 to 20 wt %, preferably 3 to 15 wt % and more preferably 5 to 10 wt %, based on the total composition.

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As well as the crystalline aluminosilicate builders already mentioned, other inorganic or organic builders may be present. Inorganic builders that may be present. Inorganic builders that may be present include sodium carbonate,
5 layered silicate, amorphous aluminosilicates, and phosphate builders, for example, sodium orthophosphate, pyrophosphate and tripolyphosphate.

Organic builders that may additionally be present include
10 polycarboxylate polymers such as polyacrylates and acrylic/maleic copolymers; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-di- and trisuccinates, carboxymethyloxysuccinates, carboxy-methyloxymalonates, dipicolinates, hydroxyethyliminodiac-
15 etates, alkyl- and alkylenylmalonates and succinates; and sulphonated fatty acid salts.

Especially preferred organic builders are citrates, suitably used in amounts of from 5 to 30 wt %, preferably from 10 to
20 25 wt %; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt %, preferably from 1 to 10 wt %.

Builders, both inorganic and organic, are preferably present
25 in alkali metal salt, especially sodium salt, form.

Detergent compositions according to the invention may also suitably contain a bleach system. The compositions of the invention may contain peroxy bleach compounds capable of
30 yielding hydrogen peroxide in aqueous solution, for example inorganic or organic peroxyacids, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulphates.

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The sodium percarbonate may have a protective coating against destabilisation by moisture. Sodium percarbonate having a protective coating comprising sodium metaborate and sodium silicate is disclosed in GB 2 123 044 (Kao).

The peroxy bleach compound, for example sodium percarbonate, is suitably present in an amount of from 5 to 35 wt %, preferably from 10 to 25 wt %.

10

The peroxy bleach compound, for example sodium percarbonate, may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt %, preferably from 2 to 5 wt %.

15

Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid precursors. An especially preferred bleach precursor suitable for use in the present invention is N, N, N', N'-tetracetyl ethylenediamine (TAED).

20

A bleach stabiliser (heavy metal sequestrant) may also be present. Suitable bleach stabilisers include ethylenediamine tetraacetate (EDTA), ethylenediamine disuccinate (EDDS), and the aminopolyphosphonates such as ethylenediamine tetramethylene phosphonate (EDTMP) and diethylenetriamine pentamethylene phosphonate (DETPMP).

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The compositions of the present invention may also include a bleach catalyst, such as manganese cyclononane derivative.

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The compositions of the present invention may also contain soil release polymers, for example sulphonated and unsulphonated PET/POET polymers, both end-capped and non-end-capped, and polyethylene glycol/polyvinyl alcohol graft
5 copolymers such as Sokalan (Trade Mark) HP22.

The compositions of the invention may also contain dye transfer inhibiting polymers, for example, polyvinyl pyrrolidone (PVP), vinyl pyrrolidone copolymers such as
10 PVP/PVI, polyamine-N-oxides, PVP-NO etc.

A powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate polymer may be included in the granular components. A preferred
15 powder structurant is fatty acid soap, suitably present in an amount of from 1 to 5 wt %.

Other materials that may be present in detergent compositions of the invention include antiredeposition
20 agents such as cellulosic polymers; fluorescers; photobleaches; inorganic salts such as sodium sulphate; foam control agents or foam boosters as appropriate; enzymes (proteases, lipases, amylases, cellulases); dyes; coloured speckles; perfumes; and fabric conditioning compounds.

25 Ingredients which are normally but not exclusively postdosed, may include bleach ingredients, bleach precursor, bleach catalyst, bleach stabiliser, photobleaches, alkali metal carbonate, water-soluble crystalline or amorphous
30 alkaline metal silicate, layered silicates, anti-redeposition agents, soil release polymers, dye transfer inhibitors, fluorescers, inorganic salts, foam control agents, foam boosters, proteolytic, lipolytic,

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amylitic and cellulytic enzymes, dyes, speckles, perfume, fabric conditioning compounds and mixtures thereof.

It is particularly preferred to include sodium carbonate.

5 This has the advantage that it helps to structure the granule, can act to control the pH of the detergent composition when dissolved and acts as a builder. Preferably 5-30% by weight sodium carbonate are present. Minor ingredients such as layering agents (for example
10 zeolite, Alusil (trade mark) or clay) may be present at a level 0.1-10%.

The present invention will be further described, by way of
15 example only, with reference to the following non-limiting Examples.

Unless stated otherwise, all quantities are parts or percentages by weight.

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EXAMPLES

In the following examples, the following test method was used to determine residues in a washing apparatus.

5

The apparatus used comprised a Miele Novotronic washing machine W916 using the Woollens 30°C setting without pre-wash. Standard powder doses of 87g were used (except where noted otherwise). The powder was dosed in the machine using
10 a dispensing device of the Lever washing ball form (UK Registered Design No. 2 031 637). This comprises a near hemispherical plastic cup with a plastic covering grid to enable filling with powder and to prevent items of clothing blocking the aperture during the wash process. The
15 dispensing device was placed on top of the cloths in the standard washing machine.

The standard load weight was 1.5kg. The load comprised 50cm x 50cm pieces of fabric as set out below.

20

Fabric	Nominal Weight (g)	No. of pieces
Black cotton poplin	29.5	13
Black polyester cotton	28.4	13
Black cotton knit	28.1	13
Commercial sulphur green	80.5	3
Sulphur green 3 ex UMIST	31.3	6

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After washing and drying, each article was first assessed visually in terms of the presence or absence of any residue and presence or absence of individual particles, residue patches and residue films, expressed as the percentage or
5 number of cloths affected.

All articles were assessed for bleach damage against a three point scale of low, medium and high intensity expressed as the percentage of cloths affected.

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Examples 1 and 2, Comparative Example AZeolite-built powders containing primary alcohol sulphate (PAS) and nonionic surfactant

5

Granules and detergent base powders were prepared as follows.

10 Nonionic surfactant granule N1

A mixture of sodium sulphate, sodium carbonate and Sokalan (Trade Mark) CP5 (acrylic/maleic copolymer ex BASF) were spray-dried to form a porous powder with the following composition:

15

Ingredients	Level (wt%)
Na ₂ SO ₄	64.2
Na ₂ CO ₃	24.0
Sokalan CP5 (ex BASF)	9.8
Water	2.0

The slurry was made by successively dosing Sokalan CP5, sodium sulphate and sodium carbonate in water. The moisture content of the slurry was 55% and the temperature 90°C. The slurry was sprayed in a countercurrent spray-drying tower using an inlet temperature of 350-400°C. Nonionic surfactant was sprayed onto this spray-dried carrier in a rotating pan granulator, resulting in the following composition:

25

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Ingredients	N1 (wt%)
Na ₂ SO ₄	45.8
Na ₂ CO ₃	17.1
Sokalan CP5 (ex BASF)	7.0
Water	1.4
Imbentin 6.5EO (ex Kolb)	28.6

5 Anionic Surfactant Granule A1

Primary alcohol sulphate (PAS) paste containing 70% neutralised cocoPAS and 30% water was dried in a dryer/granulator supplied by VRV SpA, Italy, as follows.

- 10 The temperature of the material fed into the drying zone was set at 60°C and a small negative pressure was applied to the drying zone. A throughput in the flash drier of 120 kg/hr of paste was used. The temperature of the wall of the drying zone was initially 140°C. The heat transfer areas of
15 the drying and cooling zones were 10 m² and 5m² respectively. The temperature of the wall of the drying zone was raised in steps to 170°C. Correspondingly, the throughput was increased in steps to 430 kg/hr at 170°C. The particles then passed to a cooling zone operated at a temperature of
20 30°C.

This resulted in granules with the following composition.

- 24 -

Ingredients	A1 (wt%)
coco PAS	90
Water	5
sodium sulphate/alkane	5

Builder Granules B1 and B2

- 5 The builder granules used were commercially available:
 B1: granular sodium citrate dihydrate (ex ADM)
 B2: layered silicate granules (SKS-6 ex Hoechst).

10 Detergent base powder F1 (for Comparative Example A)

- The following detergent powder formulation was processed using a Lödige mixer CB30, in which the various ingredients were mixed together, followed by a densification step in a Lödige mixer KM300. The process was substantially as
 15 described in EP 420 317A (Unilever).

Ingredients	F1 (wt%)
sodium PAS	14.2
coco ethoxylate 7EO	9.1
coco ethoxylate 3EO	6.1
Zeolite MAP (anh)	47.9
Soap	2.4
Light soda ash	2.8
SCMC	1.4
Sodium citrate (dihydrate)	7.9
water etc.	8.2

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The PAS was introduced into the Lödige CB30 as PAS powder. This powder consisted of 45% by weight of PAS, zeolite MAP and carbonate and was prepared in a Lodige CB30 by neutralisation of PAS acid with fine sodiumcarbonate
5 together with zeolite MAP under high shear. The PAS powder was continuously dosed into the CB30 together with the zeolite MAP, SCMC, citrate and light soda ash. A mixture of the ethoxylates and fatty acid was dosed into the Lodige CB30, as well as 50% NaOH solution, which neutralised the
10 fatty acid. A CB30 speed of 1500 rpm was applied. The powder exiting the CB30 was layered with zeolite MAP and brought in the KM300 where it was mixed under moderate shear. Due to the temperature/moisture content of the detergent composition in the KM300, the powder composition
15 was deformable and densification accordingly took place.

Detergent compositions 1, 2 and A

20 Fully formulated detergent powder compositions were prepared, to the formulations shown in the Table below (wt% in mixture), by mixing the various ingredients described above and postdosing other ingredients as shown. The bulk density in all cases exceeded 600 g/l.

25

Residues scores and bleach damage scores were determined as described above and are also shown below.

The compositions of the invention showed very low patch,
30 particle and filming scores and low bleach damage scores, particularly when compared with comparative Example A. Clearly, the compositions of the present invention show a marked improvement on the comparative example.

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Examples 1 and 2, Comparative Example A:
formulations and results

5

Powder	Example 1 Invention	Example 2 Invention	Example A Comparative
Base powder F1			66.7
Nonionic granules N1	22.2	22.2	
PAS granules A1	12.4	12.4	
Citrate granules B1	32.1		
SKS-6 granules B2		32.1	
TAED granules	6.7		
Sodium percarbonate	21.0		
antifoam/fluorescer granules	4.1		
Sodium bicarbonate granules	1.0		
Dequest 2047	0.4		

Ex.	Residue scores [% of articles]				Bleach damage scores [% of articles]			
	Patch es	Part- icles	Film -ing	Total	Low	Medium	High	Total
1	0	0	1	1	3	5	0	7
2	0	2	5	7	3	5	0	7
A	3	37	33	43	8	14	3	25

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Examples 3 to 5, Comparative Example BZeolite-built powders with PAS and nonionic surfactant

The following granules were used:

5

Nonionic surfactant granules N1

as described in Examples 1 and 2

Anionic surfactant granules A110

as described in Examples 1 and 2

Builder granules B3

A slurry of zeolite MAP and Sokalan CP5 (ex BASF) was spray-dried resulting in a powder with the following composition:

15

Ingredients	B3 (wt%)
Zeolite MAP	75.6
Sokalan CP5	18.9
Water	5.5

Builder granules B4 and B5

20 Zeolite MAP and sodium citrate dihydrate were dosed in a high speed granulator (Fukae FS30). To this powder mixture, a 40% Sokalan CP5 (ex BASF) solution was added and granulation was continued until a product with good granulometry was obtained. The powder was dried in a fluid bed, which resulted in a powder with the following
25 formulation (levels in wt %).

- 28 -

Ingredients	Builder B4	Builder B5
Zeolite MAP (anh)	45.4	56.3
sodium citrate 2aq	30.3	14.1
Sokalan CP5	13.0	15.5
water etc.	11.3	14.1

Detergent base powder F2 (for Comparative Example B)

- 5 The following detergent powder formulation was processed using a Fukae FS 30 granulator. The solid ingredients (zeolite, 45% PAS granules, sodiumcitrate, SCMC, soda ash) were dosed in the mixer and premixed for 20 seconds. The ethoxylates and fatty acid were premixed in a separate
- 10 vessel at a temperature of 60°C. 50% NaOH solution was added to this mixer to saponify the fatty acid, after which the mixture was quickly dosed in the granulator. Granulation was carried out using an agitator speed of 200 rpm and a chopper speed of 3000 rpm, until a satisfactory
- 15 particle size was obtained.

Ingredients	F2 (wt%)
sodium PAS	16.11
coco ethoxylate 6.5EO ¹	5.86
coco ethoxylate 3EO ¹	7.32
Zeolite MAP (anh)	49.46
Soap	2.38
Light soda ash	3.53
SCMC	1.35
Sodium citrate 2aq	6.53
water etc.	7.45

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The nonionic surfactants were Imbentin (Trade Mark) (ex Kolb).

The following fully formulated powders were prepared
5 (amounts in parts by weight):

Powder	3	4	5	B
Base powder F2				56.5
Nonionic granules N1	18.8	18.8	18.8	
PAS granules A1	10.5	10.5	10.5	
Builder granule B3	27.2			
Builder granule B4		27.2		
Builder granule B5			27.2	
TAED granules	5.7			
sodium percarbonate	17.5			
antifoam granules	3.5			
Dequest 2047	0.35			

10 The bulk density in all cases exceeded 600 g/l.

The following results were obtained in the washing experiment described above.

- 30 -

Powder	Residue scores [% of articles]			
	Patches	Particles	Filming	Total
3	0	5.2	10.8	12.5
4	0	12.5	13.0	19.3
5	0	15.6	21.4	28.1
B	14.6	25.0	37.5	45.8

Powder	Bleach damage scores [% of articles]			
	Low	Medium	High	Total
3	0	0	0	0
4	0.5	0.5	0	1.0
5	0.00	1.6	0	1.6
B	1.0	2.1	4.2	8.3

5 The compositions 3, 4, 5 and B have substantially the same total surfactant level and type: however, in the Examples 3, 4 and 5 of the present invention, the builder, anionic surfactant and nonionic surfactant were substantially separated into separate particles, whereas in Comparative

10 Example B these ingredients were together in a single base powder.

Examples 3, 4 and 5 according to the present invention showed comparatively low patch, particle and filming scores

15 when compared to Comparative Example B.

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Examples 6 and 7, Comparative Example CPhosphate-built powders containing linear alkylbenzene
sulphonate (LAS) and nonionic surfactant

5

Granules were prepared as follows:

Nonionic surfactant granules N2

10 A granule was produced using the method of Granule N1,
having the following composition:

Ingredients	N2 (wt %)
Na ₂ SO ₄	46.7
Na ₂ CO ₃	17.5
Sokalan CP5 (ex BASF)	7.1
Synperonic A7	14.3
Synperonic A3	10.7
water etc	3.8

Anionic surfactant granules A2

15 Linear alkylbenzene sulphonate (LAS) granules were produced
in a dryer/granulator from VRV SpA, Italy. LAS acid was
neutralised with sodium carbonate as follows.

Sodium linear alkyl benzene sulphonate particles (NaLAS)
were produced by neutralising LAS acid with sodium carbonate.
20 Furthermore, zeolite MAP was dosed as a layering agent was
dosed as well. A 1.2 m² VRV flash-drier machine was used
having three equal jacket sections. Dosing ports for
liquids and powders were situated just prior to the first
hot section, with mid-jacket dosing ports available in the
25 final two sections. Zeolite was added via this port in the

- 32 -

final section. An electrically-powered oil heater provided the heating to the first two jacket sections. Ambient process water at 15°C was used for cooling the jacket in the final section. Make-up air flow through the reactor was controlled between 10 and 50 m³/kg hr by opening a bypass on the exhaust vapour extraction fan. All experiments were carried out with the motor at full-speed giving a tip speed of about 30 m/s. Screw-feeders were calibrated to dose sodium carbonate and zeolite MAP for layering. The sodium carbonate and liquids were added just prior to the first hot section and zeolite layering was added into the third section which was cold. The minimum level of zeolite was added to give free-flowing granules leaving the drier.

A jacket temperature of 145°C was used in the first two sections, with an estimated throughput of components 60-100 kg/hr. A degree of neutralisation of alkyl benzene sulphonate of >95% was achieved. The bulk density, surfactant level and compressibility of the particles was then measured. They had the following composition:

Ingredients	A2 (wt %)
sodium LAS	90.0
zeolite 4A (anh)	9.0
water etc.	1.0

Builder granules B6

Sodium tripolyphosphate (STP) powder was continuously fed into a Schugi Flexomix Granulator, whilst spraying on a 10% alkaline sodium silicate solution. The material exiting the

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granulator was cooled in a fluidised bed, resulting in a granular powder with the following composition:

Ingredients	B6 (wt %)
STP	85
Sodium silicate	2.3
Water	12.7

5 Builder granule B7

Compacted STP, Rhodiaphos LV ex Rhône-Poulenc, was used as a builder granule.

Detergent base powder F3 (comparative)

- 10 A mixture of surfactants, builder, other wash active ingredients and water was spray-dried, resulting in the following composition:

Ingredients	F3 (wt %)
sodium LAS	10.59
Synperonic A7	7.06
Synperonic A3	5.30
Sokalan CP5	1.91
STP	40.19
fatty acid/soap	1.01
sodiumsulphate	7.42
SCMC	0.90
sodiumsilicate	10.59
water etc.	15.03

- 15 These granules were mixed and post-dosed to give the following formulations:

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Composition	C comparative	6 invention	7 invention
LAS granule A2		6.7	6.7
NI granule N2		28.1	28.1
Builder granule B6		26.7	
Builder granule B7			26.7
Base powder F3	54.56		
dense sulphate	22.47	11.9	11.9
Nabion 15 (ex Rhône-Poulenc)		7.7	7.7
Perborate tetrahydrate	14.00	15.1	15.1
SCMC		0.2	0.2
TAED	2.49	2.6	2.6
Antifoam/fluorescer granule	1.00	1.2	1.2
Sodium carbonate	4.10	0.1	0.1
Enzymes, perfume etc.	1.38	0.0	0.0
Bulk density [g/l]	720	765	850

	Residues [% of articles]				Bleach Damage [% of articles]
	Patches	Parti- cles	Filming	Total	Low
C	0	10.8	10.0	14.6	4.2
6	0	4.2	4.8	7.5	2.1
7	0	2.7	2.7	7.5	0

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Compositions C, 6 and 7 are similar in terms of surfactant level and surfactant type. However, in compositions 6 and 7 according to the invention, the surfactant components have been separated into separate particles and have been
5 separated from builder components. As a result, it is found that residue and bleach damage are surprisingly reduced.

10 Examples 8 and 9, Comparative Example D

Zeolite-built powders containing LAS and nonionic surfactant

The following granules were produced:

15

Nonionic surfactant granules N3

A mixture of carbonate, bicarbonate and Sokalan CP5 was spray-dried. Onto the resulting powder a mixture of alcohol ethoxylates (3EO and 7EO) was sprayed in a pan granulator,
20 resulting in the following total formulation:

Ingredients	N3 (wt%)
NaHCO ₃	28.5
Na ₂ CO ₃	35.9
Sokalan CP5	7.3
water	1.5
Synperonic A7 (ex ICI)	17.4
Synperonic A3 (ex ICI)	9.4

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Anionic surfactant granules A3

The process of Granule A2 was repeated, using a 2m² VRV machine, to produce granules containing 71.4% by weight LAS.

5 Builder granules B4 were as described above.

Detergent powder F4 (for Comparative Example D)

The following detergent powder formulation was processed using a Lödige CB30, in which the various ingredients were
10 mixed together, followed by a densification step in a Lödige KM300.

Ingredients	F4 (wt%)
sodium LAS	14.6
Synperonic A7	7.7
Synperonic A3	4.1
Zeolite MAP (anh)	46.7
fatty acid	1.9
Light soda ash	12.4
SCMC	0.9
soil release polymer	1.6
water etc.	10.1

These various ingredients were assembled into the following
15 fully formulated powders:

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Powder	8 Invention	9 Invention	D Comparative
Detergent powder F4			62.86
Nonionic granules N3	26.40	24.45	
LAS granules A3	12.30	11.40	
Builder granules B4	13.43	27.80	
TAED granules	6.75	6.00	6.00
Sodiumpercarbonate	23.25	20.00	22.50
antifoam/fluorescer granules	4.00	4.00	4.00
Sodium carbonate granules	5.34		
Sodium bicarbonate granules	1.00	1.00	1.00
Nabion 15 (ex Rhône- Poulenc)	3.00	2.50	
Dequest 2047	1.00	1.00	1.34
Savinase	0.78	1.00	1.00
Lipolase	0.25	0.25	0.30
Bulk density [g/l]	730	755	850

The residue results (dosage in machine 70 g per wash) were
5 as follows:

Powder	Residue scores [% of articles]			
	Patches	Particles	Filming	Total
8	1.0	10.9	15.6	19.3
9	1.6	14.1	19.3	24.5
D	4.7	22.9	28.6	39.1

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The compositions 8, 9 and D have substantially the same composition in terms of active level and type. However, in Examples 8 and 9 according to the invention, the nonionic surfactant, anionic surfactant and builder components are substantially separated into separate granules. As a result, marked improvements in patch, particle and film scores can be observed.

10

Examples 10 and 11, Comparative Examples E, F and G
Zeolite-built powders containing LAS and nonionic surfactant

15 The following granules were prepared.

Builder granule B8

A builder granule was produced by continuously dosing zeolite MAP, granular trisodium citrate and 40% Sokalan CP5 solution into a Lödige CB30 recycler. The CB30 was operated at 1500 rpm. The exiting powder was led through a Lödige KM300 ploughshare (120 rpm), in which densification took place. The resulting powder was dried in a fluid bed with an air temperature of 110 °C. The composition of the resulting builder granule was:

Ingredients [wt%]	B8
Zeolite MAP (anh)	41.6
Trisodium citrate	31.3
Sokalan CP5	12.2
Water etc.	14.9

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Nonionic surfactant granules N4

The nonionic surfactant granule N4 was produced using silica (Sorbosil TC15 ex Crosfield) as the carrier. It was prepared in a Fukae FS30 mixer. The following procedure was used.

- 5 Silica was dosed into the Fukae and a mixture of nonionic and fatty acid, heated to approximately 60°C, was added to the solids, after which a 50% NaOH solution was sprinkled on top. Directly after addition of the NaOH, the mixture was granulated, using agitator speeds of 100-200 rpm and a
10 chopper speed of 3000 rpm. Typical granulation time was 1 min. The resulting powder was layered with silica and removed from the granulator. The composition of nonionic granule I was the following:

15

Ingredients	N4
Sorbosil TC15	26.1
Neodol 91-6	64.7
Soap	7.8
Water etc.	1.4

Nonionic surfactant granules N5

- These granules were produced by first spray-drying a mixture
20 of carbonate, bicarbonate, citrate and Sokalan CP5. The spray-dried material was dosed into a Lödige FM300 D after which nonionic was sprayed on. The Lödige was operated at a speed of 120 rpm with the choppers switched off. Spray on was carried out for 12 minutes. The final composition was as
25 follows:

- 40 -

Ingredients	N5
Synperonic A7	25.4
NaHCO ₃	31.8
Na ₂ CO ₃	31.8
Sokolan CP5	8.0
Water, minors etc.	3.0

Nonionic surfactant granule NX (for Comparative Example E)

5 was made by using the spray-dried carrier as described. In this case, the spray-dried carrier was mixed with zeolite MAP, after which nonionic was mixed in and granulation was carried out in an Eirich RV02 mixer. The Eirich was operated with a stirrer speed of 400 rpm. Granulation was carried out
 10 for 10 seconds. The final composition was as follows:

Ingredients	NX
Synperonic A7	26
NaHCO ₃	16.6
Na ₂ CO ₃	16.6
Zeolite MAP (anh)	31.5
Sokolan CP5	4.2
Water, minors etc.	5.1

As can be seen the zeolite level in this nonionic granule is clearly above the maximum level of 10% specified according
 15 to the present invention.

Anionic surfactant granules A4 and A5

These was prepared by the method used for Granule A2.

Anionic surfactant granule A4 had the following composition:

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Ingredients	A4
NaLAS	81.0
Zeolite MAP (anh)	10.0
Carbonate	5.0
Water, NDOM etc	4.0

Anionic surfactant granule A5 was made in the same manner, using a 2m² VRV machine, but had a NaLAS content of 70 wt%,
5 and contained 20 wt% zeolite 4A and 5 wt% zeolite MAP.

Anionic surfactant granule AX (for Comparative Example F)
was prepared by continuously dosing LAS acid, sodium carbonate and zeolite MAP in a Lödige CB30 recycler. The
10 product was granulated in the CB30 and cooled in the fluid bed to obtain free flowing granules. The composition of anionic surfactant granule AX was as follows:

Ingredients [wt%]	AX
NaLAS	47.1
Zeolite MAP (anh)	36.0
Carbonate	5.6
Water, NDOM etc	11.2

15 The NaLAS level is lower than the 60% minimum specified in accordance with the present invention.

Detergent powders were prepared by mixing to the formulations shown below. Examples 10 and 11 are in
20 accordance with invention, Examples E, F and G are comparative. For Comparative Example G the base powder F4 was used.

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Formulation [wt%]	10	E	F	11	G
Granule B8	22.6	23.2	17.7	22.0	
Granule N4				10.8	
Granule N5	27.5		27.5		
Granule NX		26.9			
Granule A4				10.7	
Granule A5	12.4	12.4			
Granule AX			17.3		
Base powder F4					61.0
Percarbonate	19	19	19	19	19
TAED	5.5	5.5	5.5	5.5	5.5
EAG adjunct	1.7	1.7	1.7	1.7	1.7
SCMC	0.5	0.5	0.5	0.5	
Fluorescer adjunct	1.3	1.3	1.3	1.3	1.3
PVP	0.1	0.1	0.1	0.1	0.1
Soil release polymer granule	1.5	1.5	1.5	1.5	1.5
Sokolan CP5 granules					1.0
Nabion 15	5.5	5.5	5.5	5.5	5.5
Dense carbonate	0.5	0.5	0.5	19.5	0.5
Sodiumbicarbonate					1.0
Dequest 2047	1	1	1	1	1
Savinase 12.0T	0.78	0.78	0.78	0.78	0.78
Lipolase 100 T	0.12	0.12	0.12	0.12	0.12
BD [g/l]	843	852	799	694	893

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The residue scores are shown below. Clearly the invention products performed better than the comparative examples.

Product	Dye damage scores (total) [% of cloths]	Residue scores (total) [% of cloths]
10 (Invention)	2.1	28.6
E (Comparative)	4.7	43.2
F (Comparative)	5.7	32.8
11 (Invention)	0.7	14.5
G (Comparative)	5.7	50.0

5

If the zeolite level in the nonionic granule is too high, a high level of residues and dye damage is observed (Comparative Example E).

10

Similarly if the active level in the anionic granule is too low, the dye damage will be high (Comparative Example F).

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Examples 12 and 13

Builder granules B8, nonionic surfactant granules N5 and
5 anionic surfactant granules A1 and A5 were as described in
Examples 10 and 11.

Nonionic surfactant granule N6 was prepared by a process
route consisting of a Lödige CB30, followed by a Niro fluid
10 bed and a Mogensen sieve. The Lödige CB30 was operated at
1500 rpm. Water was used to cool the CB30 jacket during the
process. The air flow in the Niro fluid bed was 900-1000
m³/hr. The total flow of powder exiting the process was in
the order of 600 kg/h.

15 Silica (Sorbosil (Trade Mark) TC15 ex Crosfield) was
continuously dosed into the CB30, into which also a mixture
of nonionic surfactant (Lutensol AO7 ex BASF) and fatty acid
(Pristerene 4916 ex Unichema) was dosed via dosing pipes. At
20 the same time 50% NaOH was dosed to neutralise the fatty
acid. This set of solid and liquid materials was mixed and
granulated in the CB30 after which the resulting powder was
entered in the fluid bed and cooled with ambient air. Fines
were filtered from the air stream with a cyclone and filter
25 bags. Coarse particles (>1400µm) were separated from the
product by the Mogensen sieve.

Composition [wt%]	N6
Sorbosil TC15	30.0
Lutensol AO7	55.0
Soap	13.1
Water	1.9

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These granules were mixed with other postdose materials to make products according to the invention:

Formulation	12	13
Builder granule B8		10
LAS granule A5	26	
PAS granule A1		17.8
Nonionic granule N5	38.6	
Nonionic granule N6		29
Granular citrate		7.6
Dense carbonate	1.8	2
Percarbonate	19.00	19.00
TAED	5	5
EAG adjunct	1.7	1.7
SCMC	0.6	0.6
Fluorescer adjunct	1.3	1.3
Nabion 15	5	5
Dequest 2047	1	1
Total surfactant [%]	28.0	35.9
BD [g/l]	750	682
DFR [ml/s]	118	134

CLAIMS

- 1 A particulate detergent composition or component having
5 a bulk density of at least 600 g/l and comprising at least
10% by weight of organic detergent surfactant and from 10 to
70% by weight of detergency builder, characterised in that
the composition is composed of at least two different
granular components:
- 10 (i) granules comprising at least 60% by weight of anionic
surfactant, and
- (ii) granules comprising at least 20% by weight of nonionic
15 surfactant, and less than 10% by weight of
aluminosilicate.
- 2 A detergent composition or component as claimed in
20 claim 1, characterised in that it contains from 1 to 50% by
weight of the nonionic surfactant granules (ii).
- 3 A detergent composition or component as claimed in
25 claim 2, characterised in that it contains from 1 to 30% by
weight of the nonionic surfactant granules (ii).
- 4 A detergent composition or component as claimed in any
30 preceding claim, characterised in that it contains from 1 to
70% by weight of the anionic surfactant granules (i).

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5 A detergent composition or component as claimed in any
preceding claim, characterised in that it further comprises
granules (iii) comprising up to 100% by weight of detergency
builder and optionally from 0 to 10% by weight of anionic
5 and/or nonionic surfactant.

6 A detergent composition or component as claimed in
claim 5, characterised in that the builder granules (iii)
10 comprise up to 90% by weight of builder and optionally from
0 to 10% by weight of anionic and/or nonionic surfactant.

7 A detergent composition or component as claimed in
15 claim 5 or claim 6, characterised in that it comprises from
5 to 80% by weight of the builder granules (iii).

8 A detergent composition or component as claimed in any
20 preceding claim, characterised in that it comprises from 15
to 70 wt% of builder.

9 A detergent composition or component as claimed in any
25 preceding claim, characterised in that the builder is alkali
metal aluminosilicate.

10 A detergent composition or component as claimed in any
30 preceding claim, characterised in that the granules (i)
comprising at least 60% by weight of anionic surfactant
optionally comprise from 0 to 40% by weight of detergency
builder.

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11 A detergent composition or component as claimed in any preceding claim, characterised in that the granules (i) containing at least 60% by weight of anionic surfactant comprise less than 15% by weight of aluminosilicate builder.

5

12 A detergent composition or component as claimed in any preceding claim, characterised in that the granules (ii) containing at least 20% by weight of nonionic surfactant
10 comprise nonionic surfactant carried on water-soluble carrier material, the nonionic surfactant being present at a level of 20 to 50% by weight based on the granules (ii).

15 13 A detergent composition or component as claimed in any one of claims 1 to 11, characterised in that the granules (ii) containing at least 20% by weight of nonionic surfactant comprise nonionic surfactant carried on a water-insoluble carrier material comprising silica or
20 aluminosilicate or a mixture thereof.

14 A detergent composition or component as claimed in any preceding claim, characterised in that the total quantity of
25 detergent surfactant in the composition is at least 12% by weight.

15 A detergent composition or component as claimed in any
30 preceding claim, characterised in that it comprises from 1 to 40% by weight of nonionic surfactant.

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- 16 A particulate detergent composition or component having
a bulk density of at least 600 g/l and comprising at least
10% by weight surfactant and from 15 to 70% by weight of
builder, characterised in that it is composed of at least
5 three different granular components:
- (i) granules comprising at least 60% by weight of anionic
surfactant,
- 10 (ii) from 1 to 30% by weight of granules comprising at least
20% by weight of nonionic surfactant, and less than 10%
by weight of aluminosilicate,
- (iii) granules comprising up to 90% by weight of builder and
15 from 0 to 10% by weight of anionic or nonionic
surfactant.

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17 A method of manufacturing a detergent powder composition or component as claimed in claim 1, comprising the steps of:

5 (i) manufacturing granules comprising at least 60% by weight of anionic surfactant,

(ii) manufacturing granules containing at least 20% by weight nonionic surfactant and less than 10% by weight
10 aluminosilicate,

(iii) optionally manufacturing granules comprising up to 100% by weight of builder and optionally from 0 to 10% by weight of nonionic or anionic surfactant, and

15 mixing the granules produced in steps (i) and (ii), and optionally the granules produced in step (iii).

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INTERNATIONAL SEARCH REPORT

Intern al Application No
PCT/EP 98/02982

A. CLASSIFICATION OF SUBJECT MATTER

C 11 D 1/83, C 11 D 17/06

According to International Patent Classification (IPC) or to both national classification and IPC6

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C 11 D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0451894 A1 (UNILEVER NV) 16 October 1991 (16.10.91), the whole document. --	1-16
A	WO 96/38530 A1 (HENKEL KGGA) 05 December 1996 (05.12.96), page 17, lines 14-30, examples, claims. --	1-8, 10-12, 14-16
A	EP 0550086 A1 (UNILEVER N.V.) 07 July 1993 (07.07.93), claims 1,3,4. -----	1-8, 10-12, 14-16

☐ Further documents are listed in the continuation of box C.☐ Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
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- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- * & * document member of the same patent family

Date of the actual completion of the international search

25 September 1998

Date of mailing of the international search report

27. 10. 98

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ANHANG

zum internationalen Recherchen-
bericht über die internationale
Patentanmeldung Nr.

ANNEX

to the International Search
Report to the International Patent
Application No.

ANNEXE

au rapport de recherche inter-
national relatif à la demande de brevet
international n°

PCT/EP 98/02982 SAE 196032

In diesem Anhang sind die Mitglieder
der Patentfamilien der im obenge-
nannten internationalen Recherchenbericht
angeführten Patentdokumente angegeben.
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This Annex lists the patent family
members relating to the patent documents
cited in the above-mentioned inter-
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membres de la famille de brevets
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dans le rapport de recherche inter-
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