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(54) Title: DETERGENT COMPOSITIONS CONTAINING NONIONIC SURFACTANT GRANULE

(57) Abstract

A particulate detergent composition is composed of at least two different granular components, one of which is a nonionic-surfactant-containing granular component characterised in that it comprises more than 55 % by weight of nonionic surfactant, at least 5 % by weight of silica having an oil absorption capacity of at least 1.0 ml/g and, optionally, less than 10 % by weight of aluminosilicate. Preferably, the composition also contains a spray-dried or agglomerated detergent base powder containing surfactant and builder, or a granule containing a high concentration of anionic surfactant, a builder granule, or any combination of these.

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DETERGENT COMPOSITIONS CONTAINING NONIONIC SURFACTANT GRANULE

Technical field

The present invention relates to particulate laundry detergent compositions which include nonionic-surfactant-containing granular compositions.

Background

It is frequently desired to include nonionic surfactant in particulate laundry detergent compositions as it gives good oily soil detergency and can reduce foam levels, which is beneficial in detergent compositions for use in automatic washing machines.

Particulate laundry detergent compositions are typically produced by the spray-drying process or by an agglomeration process. These processes are well known to the skilled person and have their own particular problems and advantages. There may be problems during manufacture of compositions having nonionic surfactant in spray drying processes due to breakdown of nonionic surfactant leading to the emission of smoke. Problems of poor dispersion in wash water have been encountered with granular detergents manufactured in agglomeration processes, which may be due to unfavourable interactions between nonionic surfactant and other detergent components.

For these reasons, it is desired to add nonionic surfactant to granular detergent compositions made by either process after the granulates are formed. In particular, there is now interest in adding particles containing nonionic surfactant to such granular compositions. As most nonionic surfactants are liquids or waxy solids, they need to be borne on a carrier.

Nonionic-surfactant-containing particles are disclosed for example in JP 08 027498A (Kao), which discloses a silica-based carrier having an oil absorption capacity of at least 80 ml/g and capable of providing a particle having up to 50% by weight of nonionic surfactant. It is desired however to provide a greater carrying capacity.

JP 07 268 398A (Lion) discloses a nonionic surfactant containing granular composition having up to 70 % by weight of nonionic surfactant and less than 5% by weight silica. However, such granules contain a quantity of aluminosilicate. The inventors have discovered unfavourable interactions between nonionic surfactant and aluminosilicates leading to poor dispersion if large quantities of aluminosilicate are present. Further, where large quantities of nonionic surfactant are included, there is frequently a problem of leaching out of the surfactant from the composition in storage. There may also be problems of low particle strength.

Summary of the invention

The present inventors have now discovered that nonionic-surfactant-containing granules can be manufactured having a high content of nonionic surfactant and containing low quantities of aluminosilicate, the particles showing low leaching tendency and good strength.

The present invention is concerned with a particulate detergent composition including a specific granular component: a nonionic-surfactant-containing granular composition comprising more than 55% by weight of nonionic surfactant, at least 5% by weight of silica having an oil absorption capacity of at least 1.0 ml/g and, optionally, less than 10% by weight of aluminosilicate.

Accordingly, the present invention provides a particulate detergent composition composed of at least two different granular components:

(a) a nonionic-surfactant-containing granular component comprising:

  (a1) more than 55% by weight of nonionic surfactant,

  (a2) at least 5% by weight of silica having an oil absorption capacity of at least 1.0 ml/g,

  (a3) optionally, less than 10% by weight of aluminosilicate,

(b) at least one other granular component.

The nonionic-surfactant-containing granule

Preferably, the high-nonionic granule comprises at least 59% by weight of nonionic surfactant.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially C_8-C_{20} aliphatic alcohols ethoxylated with an average of 1-20 moles of ethylene oxide per mole of alcohol, and more especially the C_9-C_{15} primary and secondary aliphatic alcohol ethoxylated with an average of from 1-10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkyl polyglycosides, glycerol monoethers, and polyhydroxy amides (glucamide).

The inventors have discovered that it is necessary to use at least 5% by weight of silica having an oil absorption capacity of at least 1.0 ml/g. Oil absorption capacity is a parameter which is well known and can be measured by the technique described in DIN ISO 787/5. Preferably, the oil absorption capacity is at least 1.5 ml/g, more preferably at least 2.0 ml/g and most preferably at least 2.5 ml/g.

Preferably, the granule contains at least 10%, more preferably at least 15%, of silica.

Silica having the required oil absorption capacity is commercially available.

Preferably, if the high-nonionic granule contains aluminosilicate, less than 5% by weight is present.

Crystalline aluminosilicates (zeolites) are preferred.

Aluminosilicates are materials having the general formula:

$$0.8\text{-}1.5\ M_2O.\ \ Al_2O_3.\ \ 0.8\text{-}6\ \ SiO_2$$

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. The preferred sodium aluminosilicates contain 1.5-3.5 SiO_2 units in the formula above. They can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

The zeolite which may be used in the nonionic-surfactant-containing granules 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 invention, the zeolite incorporated in the nonionic-surfactant-containing granules 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) MAP from Crosfield Chemicals Ltd, UK.

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. 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.

The nonionic-surfactant-containing granules of the present invention may contain other material. In particular, the granules may contain a structurant, which may also be considered as a binder, in order to improve the strength of the granules.

The granules may contain about 2 to 15% by weight of a structurant. Suitable structurants include, for example, soaps, sugars, succinates, silicates, citrates, or polymers such as polyethylene/propylene glycol of molecular weight 1000 to 12 000, polyacrylate of molecular weight 30 000 to 200 000, polyvinyl alcohol of molecular weight 30 000 to 200 000, or acrylate/maleate copolymers, eg Sokalan (Trade Mark) CP5 ex BASF.

Especially preferred structurants are selected from the following list: polyethylene glycol, soap, maltose, glucose, sucrose, polyvinyl alcohol, and acrylate/maleate copolymer in admixture with glucose, sodium chloride or trisodium citrate.

Other minor ingredients such as water may be present, at a level of preferably less than 5% by weight.

The granules may optionally contain from 0 to 5% of anionic surfactant, such as alkyl benzene sulphonates, particularly linear alkyl benzene sulphonates having an alkyl chain length of C_8-C_{15}, primary and secondary alkyl sulphates, particularly C_8-C_{15} primary alcohol sulphates, alkyl ether sulphates, olefin sulphonates, alkyl xylene sulphonates, dialkyl sulphosuccinates and fatty acid ester sulphonates. Optionally, layering agents such as layered silicate and/or zeolite may be included at a level of about 0 to 10 % by weight as long as the total quantity of zeolite remains below 10% by weight.

The nonionic-surfactant-containing granules of the present invention preferably have a bulk density in the range of from 400 to 800 g/l. The granule sizes are preferably in the range of from 200 to 1000 micrometres.

Preparation of the nonionic-surfactant-containing granules

The nonionic-surfactant-containing granules are manufactured by any suitable method. Preferably, the components are granulated together in a mechanical mixer. Preferably, a high-speed mixer/densifier or granulator is used.

One method comprises granulating together in a mixer greater than 55% by weight of nonionic surfactant, at least 5% by weight of silica having an oil absorption capacity of at least 1.0 ml/g, less than 10% by weight of aluminosilicate. The liquid components may be introduced by spraying them in while the mixer is running. Preferably, in this case, a relatively large quantity of structurant (5-15% by weight, preferably 5-10% by weight) is preferably used to give granules of adequate stability as measured by their nonionic surfactant leaching tendency (see below). It has been found that structurant can only be included in place of some nonionic surfactant. Accordingly, the carrying capacity of the granules is reduced compared to the theoretical maximum.

The inventors have discovered that a two-step process can be used, as a result of which less structurant is required.

Accordingly, a further subject of the present invention is a second method for the production of the nonionic-surfactant-containing granules, comprising the steps of:

(i) mixing 70-100% by weight of the solid components, 70-100% by weight of the nonionic surfactant and less than 50% by weight of the structurant, and,

(ii) adding the remainder of the nonionic surfactant, structurant and solid components and mixing further.

It is possible that some of the solid components can be added in the second step, but preferably at least 80%, more preferably at least 90% by weight of the solid components are incorporated in the first step. Preferably, at least 75% by weight of nonionic surfactant is added in the first step.

Preferably, all of the silica is included in the first step. Preferably, at least 70% by weight of the structurant is added in the second step, more preferably at least 80% by weight.

Where the structurant comprises soap, it may be produced in situ by neutralisation of fatty acid, by, for example, caustic soda or soda ash. This also applies to the one-step process.

Without wishing to be bound by theory, it is believed that the process of formation of nonionic-surfactant-containing granules proceeds as follows.

In the first step, it is believed that small particles of generally spherical shape are produced, having nonionic surfactant mainly in the pores of solid material. In the second step, it is believed that such small particles are agglomerated into larger agglomerations by the addition of further nonionic surfactant and/or structurant.

The two-step process for production of nonionic surfactant granules can be used to produce granules of stability similar to or greater than for the first-step process but having lower quantities of structurant (in the region of 2 to 10%, preferably less than 5% by weight of structurant) or particles having similar levels of structurant (from 5 to 15% by weight, preferably from 5 to 10% by weight) and having similar or greater stability as measured by nonionic leaching tendency. The first and second steps may be carried out in a high shear mixer.

In both the first and second methods described above, the components may be mixed in an Eirich Mixer, for example an Eirich RV02 Granulator. Other equipment suitable for use in the present invention include the Fukae mixer, produced by Fukae Powtech Co. of Japan, the Diosna V Series supplied by Dierks & Sohne Germany, the Pharma Matrix ex TK Fielder Ltd England, the Fuji V-C Series produced by Fuji Sangyo Company Japan and the Roto produced by Zanchetta & Company Srl, Italy. Other suitable equipment can include the Lödige Series CB for continuous high shear granulation available from Morton Machine Company Scotland, the Drais T160 Series manufactured by Drais Werke GmbH, Mannheim Germany.

High shear mixing can be achieved by the skilled person in a manner well known in the art. For example, where a Lödige Mixer is used, a rotation speed of 500-3000 rpm may be used.

Other granular components

As previously indicated, the detergent compositions of the invention contain at least one other granular component in addition to the nonionic-surfactant-containing granular component (high-nonionic granule). The other component is selected from the following list:

(i) a conventional spray-dried or agglomerated base powder granule containing anionic surfactant, builder and, optionally nonionic surfactant, and/or

(ii) a builder granule, and/or

(iii) a granular component containing at least 60% by weight of anionic surfactant (high-anionic granule).

The nonionic-surfactant-containing granules can be mixed with conventional surfactant-containing base powders in order to increase the nonionic surfactant content of the overall composition. Steps such as spraying nonionic surfactant onto base powder can then be reduced or avoided. High total quantities of nonionic surfactant in the mixture can be obtained.

The nonionic-surfactant-containing granules can be mixed with conventional base powders containing little or no nonionic surfactant, or with builder granules, in order to effectively separate nonionic surfactant from aluminosilicate builder. As noted above, there is believed to be an unfavourable interaction between nonionic surfactant and aluminosilicate builder which leads to problems in dispersion.

The base powders or builder granules may be manufactured by any suitable process. For example, they may be produced by spray-drying, spray-drying followed by densification in a batch or continuous high speed mixer/densifier or by a wholly non-tower route comprising granulation of components in a mixer/densifier, preferably in a low shear mixer/densifier such as a pan granulator or fluidised bed mixer. Methods of manufacturing a high anionic-detergent-active granular component are also discussed below.

The separately produced granular components may be dry-mixed together in any suitable apparatus.

The nonionic-surfactant-containing granules may be present at a level of up to 50% by weight, preferably from 2 to 50% by weight, the other granular component or components constituting the remaining 50 to 98% by weight of the totality of granular components. The other granular components may be, for example, a base powder alone, a base powder plus another high-active granule, or a number of separate granules (eg a builder granule , a high-anionic granule).

The amount of nonionic-surfactant-containing granules is more preferably up to 40% by weight, but may be present at levels of as low as from 2 to 10% by weight.

The individual granular components may be of any suitable bulk density.

The inventors have found that, where a given formulation of detergent composition is produced by dry-mixing at least two granular components having different surfactant levels, the detergent composition has better powder properties such as stability than if the formulation were produced with all the components in a single granule.

Anionic-surfactant-containing granules

A method of producing a detergent component containing at least 60% by weight of anionic surfactant is set forth in WO 97/32002A (Unilever). The process comprises the steps of feeding a paste material comprising water and an anionic surfactant into a drying zone, heating the paste material in the drying zone to reduce the water content thereof and subsequently cooling the paste material in a cooling zone to form detergent particles, characterised by introducing a layering agent into the cooling zone during the cooling step. This process may be carried out in a machine manufactured by VRV Impianti SpA, having a heating surface area of 1.2 m^2. The heating zones are maintained at a temperature in the region of 120-190°C, for example 170°C. Cooling is achieved using ambient process water at 15°C. The apparatus is used with tip speed of the blades of 30 m/s.

A method of producing a detergent component containing at least 75% by weight of anionic surfactant is set forth in WO 96/06916A and WO 96/06917A. A paste material comprising water in an amount of more than 10% by weight of the paste and the surfactant is fed into a drying zone, the paste is heated to a temperature in excess of 130°C to reduce the water content to more than 10% by weight and the material is subsequently cooled to form detergent particles.

The granules containing anionic surfactant may suitably be present at a level of from 5 to 35% by weight, preferably from 5 to 20% by weight.

Detergent compositions

The detergent composition of the present invention may comprise only the specified granular components. In this form, it may provide a complete detergent composition for use in fabric washing or it may provide a component for a complete detergent, additional powdered components being dry-mixed with the granular component(s). The totality of the granular components is thus analogous to a conventional base powder.

Suitable components which may be post-dosed to the mixture of granular components will be discussed further below.

The mixture of granular components may be subjected to a step in which small quantities of ingredients (for example perfume) are sprayed onto the granular material.

Preferably, the totality of the specified granular components provides at least 40% by weight, preferably at least 50% by weight of the final composition, the remaining less than 60%, preferably less than 50% by weight, if present, being constituted by postdosed or sprayed-on ingredients.

In this section all percentages are based on the final composition, ie the totality of granular components, plus any sprayed-on or postdosed ingredients.

Preferably, the quantity of anionic surfactant present is in the range of from 3 to 30 % by weight of the total (final) composition. However, the invention also encompasses compositions in which the surfactant component is composed substantially wholly of nonionic surfactant. If both types of surfactant are present, the weight ratio of nonionic to anionic surfactant is preferably within the range of from 3:1 to 1:3.

The total quantity of detergent surfactant is preferably at least 10% by weight, more preferably at least 12% by weight, and most preferably at least 15% by weight. The present invention may especially be used to achieve higher surfactant loadings than may otherwise be possible, for example, greater than 20%, without loss of powder properties.

The detergent compositions of the invention also contain one or more detergency builders. The total amount of detergency builder in the compositions will suitably range from 5 to 80 wt %, preferably from 10 to 60 wt %. Builders are normally wholly or predominantly included in the granular components. Builder-containing granular components may contain less than 5% of detergent surfactant, preferably substantially no surfactant.

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

Organic builders that may additionally be present include polycarboxylate polymers such as polyacrylates and acrylic/maleic copolymers; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyl-oxymalonates, dipicolinates, hydroxyethylimino-diacetates, alkyl- and alkyenylmalonates 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 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 in alkali metal salt, especially sodium salt, form.

Detergent compositions according to the invention may also suitably contain a bleach system. It is preferred that the compositions of the invention contain peroxy bleach compounds capable of yielding hydrogen peroxide in aqueous solution, for example inorganic or organic peroxyacids, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persili-cates and persulphates. Bleach ingredients are generally post-dosed as powders.

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 %.

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 %.

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).

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).

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

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 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 PVP/PVI, polyamine-N-oxides, PVP-NO etc.

The compositions of the invention may also contain alkali metal, preferably sodium, carbonate, in order to increase detergency and ease processing. Sodium carbonate may suitably be present in amounts ranging from 1 to 60 wt %, preferably from 2 to 40 wt %. However, compositions containing little or no sodium carbonate are also within the scope of the invention. Sodium carbonate may be included in granular components, or post-dosed, or both.

The detergent composition may contain water-soluble alkali metal silicate, preferably sodium silicate having a $SiO_2:Na_2O$ mole ratio within the range of from 1.6:1 to 4:1.

The water-soluble silicate maybe 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 aluminosilicate (anhydrous basis).

Other materials that may be present in detergent compositions of the invention include antiredeposition 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.

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 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.

The present invention will be further described by way of the following non-limiting Examples.

Except where stated otherwise, all quantities are in parts or percentages by weight.

In the following examples, the following test methods will be used:

Dynamic Flow Rate (DFR)

The dynamic flow-rate or DFR is measured by the following method. The apparatus used consists of a cylindrical glass tube having an internal diameter of 35 mm and a length of 600 mm. The tube is securely champed in a position such that its longitudinal axis is vertical. Its lower end is terminated by means of a smooth cone of polyvinyl chloride having an internal angle of 15° and a lower outlet orifice of diameter 22.5 mm. A first beam sensor is positioned 150 mm above the outlet, and a second beam sensor is positioned 250 mm above the first sensor.

To determine the dynamic flow-rate of a powder sample, the outlet orifice is temporarily closed, for example, by covering with a piece of card, and powder is poured through a funnel into the top of the cylinder until the powder level is about 10 cm higher than the upper sensor; a spacer between the funnel and the tube ensures that filling is uniform. The outlet is then opened and the time t (seconds) taken for the powder level to fall from the upper sensor to the lower sensor is measured electronically. The measurement is normally repeated two or three times and an average value taken. If V is the volume (ml) of the tube between the upper and lower sensors, the dynamic flow rate DFR (ml/s) is given by the following equation:

$$DFR=V/t$$

The averaging and calculation are carried out electronically and a direct read-out of the DFR value obtained.

Average Particle Size

The mean diameter (RRd) of the particles is measured by seive analysis and calculated according to the Rosin Rammler method.

Stability measurement and nonionic leaching tendency

The stability properties were measured as follows:

(i) Paper filter storage

200g of powder was placed into a metal tin and levelled. A weighed paper filter (Schleicher & Schull, No. 589[1], diameter 11cm) was put onto this powder bed. Onto this filter another 500 gr of powder was put, followed by a second weighed filter of the specified type. Finally 200g of powder was placed onto the second filter. The tin was subsequently sealed and stored at 37°C for 1 week. After this period the tin was opened and the powder and filters removed. The filters were reweighed and the weight increase was noted. The average weight increase of the two filters specifies the absolute amount of nonionic surfactant leaching from the powder.

Another useful measure is the amount of nonionic surfactant leached out relative to the amount of nonionic surfactant present in the powder. This can be calculated as follows:

$$\text{Relative amount leached out [mg/g]} = \frac{\text{Filter weight}_{\text{after}} - \text{Filter weight}_{\text{before}}}{\text{Sample weight [g]} \times \text{Nonionic level in powder}}$$

(ii) Polyurethane filter storage

5g of powder was brought into a glass jar and levelled. Onto this powder a weighed polyamide filter (Sartolon, polyamide filter with 0.2 mm pore diameter ex Sartorius) was put. Onto this filter another 20 gr of powder was put. The jar was subsequently sealed and stored at 37°C for 1 week. After this period the jar was opened and the powder and filter removed. The filter was reweighed and the weight increase was noted. The average increase of two measurements specifies the absolute amount of nonionic surfactant leaching from the powder.

Solubility measurement

5g of the powder under investigation is dosed into 500ml of water cvontained in 1000ml beaker at a temperature of 20°C. The water is stirred with a magnetic stirring rod of 6cm maintaning a 4cm vortex for 2 minutes after which the solution is poured over a filter with a mesh size of 125 µm. The filter with residue is dried at 80°C in an oven for an hour after which the amount of residue is weighed. The amount of insolubles is calculated by:

$$\text{Insolubles [\%]} = \frac{\text{Amount of residue [g]}}{\text{Amount of initial powder [g]}} \times 100\%$$

Example 1, Comparative Example A

In this Example, a powder (Example 1) made by mixing a nonionic-surfactant-containing granule and a builder granule is compared with a powder of similar formulation (Comparative Example A) prepared as a single granulate.

Nonionic granule N1, Comparative Powder A

A nonionic-surfactant-containing granule N1 and a fully formulated powder A were made by mixing the components listed in an Eirich RV02 granulator.

Ingredients [wt%]	N1 (Invention)	A (Comparative)
SiO_2 (Sorbosil TC 15 ex Crosfield)	26.6	10.8
Nonionic (Synperonic A7)	66.1	26.6
Structurant (PEG 1500)	7.3	3.0
Zeolite MAP		20.2
Na-citrate		13.8
Light soda ash		8.9
Sokalan CP5 powder		8.9
water		7.9

Builder granule B1 was made by continuously dosing zeolite and trisodium citrate into a Lödige CB30 mixer, together with 40% Sokalan CP5 solution ex BASF. A typical speed of 1500 rpm was used. The powder was further densified in a Lödige KM 300, after which the powder was continuously dried in a fluid bed, using air with a temperature of 100-120°C. The resulting product was sieved and the fraction < 2000 μm was kept.

Ingredients	B1 (Weight %)
Zeolite MAP (anh)	41.3
Trisodium citrate	31.7
Sokalan CP5	13.8
Water	13.2

Nonionic granule N1 was dosed together with builder granule B1 and dense sodium carbonate into a V-blender and mixed to make Powder 1:

Ingredients [wt%]	Powder 1
Nonionic granule N1	39.7
Builder granule B1	49.3
Dense sodium carbonate	10.9

The following powder properties for powders A (comparative) and 1 (invention) were found:

Powder	A Comparative	1 Invention
BD [g/l]	622	723
DFR [ml/s]	110	130
BD after 1 week storage at 37°C [g/l]	621	748
DFR after 1 week storage at 37°C [ml/s]	109***	135
Rosin Rammler mean diameter [μm]	808	617
Paper filter, 1 week, [mg]	468	32
Paper filter, 1 week [mg/g nonionic in sample]	2.2	0.15
Polyamide filter, 1 week [mg]	135.9	30.3
Polyamide filter, 1 week [mg/g nonionic in sample]	20.5	4.6
Insolubles [%]	>20	4

*** Indicates that several taps were needed to induce flow from the test tube.

The above table indicates that Powder 1, according to the invention, has improved storage properties. That is, the leaching out of nonionic surfactant is very much less than with the comparative Powder A, which has a comparable overall composition and particle size distribution. Further, flow properties are improved over comparative Powder A, and Powder 1 dissolves more efficiently in the dissolution test than the comparative Example A of the same overall composition.

Examples 2 and 3, Comparative Example B

In these Examples nonionic granules within and outside the invention were combined with a builder granule to form powders.

Nonionic granules N2-N4 (invention) and NX (comparative)

Nonionic granules containing soap as structurant were prepared in a Fukae FS30 granulator. The following procedure was used.

Solid raw materials (zeolite, silica) were dosed into the granulator and pre-mixed (if applicable) for 10 seconds, using an agitator speed of 100 rpm and a chopper speed of 3000 rpm. A mixture of nonionic and fatty acid, heated to approximately 60°C was added on top of the solids, after which 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 chopper speed of 200 rpm. Typical granulation times were 0.5 to 2.5 mins. The resulting powder was layered with silica or zeolite and removed from the granulator.

The compositions of the granules are shown in the following table.

Composition [wt%]	N2	N3	N4	NX
SiO_2 (Sorbosil TC 15)	22.5	22.5	26.1	6.7
Zeolite (Wessalith P (anh))	9	9		54.8
Nonionic (Synperonic A7)	59.0			
Nonionic (Neodol 91-6)		59.0	64.7	28.3
Soap	7.1	7.1	7.8	3.4
Water etc.	2.4	2.4	1.4	6.8
BD [g/l]	605	603	582	821
DFR [ml/s]	118	126	99	114
RRd [μm]	790	685	857	896
Paper filter, 1 week, mean [mg]	57	55	55	63
Polyamide filter, 1 week, mean [mg]	54.6	98.0	94.3	95.7
Polyamide filter, 1 week [mg/g nonionic sample]	4.3	6.6	5.8	13.5
Paper filter, 1 week, mean, [mg/g nonionic in sample]	0.12	0.12	0.11	0.28

The compositions N2 to N4 according to the invention have a high nonionic surfactant level. This is due to the use of the silica carrier in place of zeolite. The compositions N2 to N4 according to the invention have a lower zeolite level than the composition NX which is comparative. They have similar particle size distributions to comparative composition NX. It is clear that the compositions according to the invention which have high silica levels and low zeolite levels generally have similar flow properties to the comparative example. Further, the product of the invention has similar or better storage properties when measured in terms of the leaching of nonionic surfactant into polyamide or paper filters. This is particularly apparent when the weight increase of the filters is given in terms of the nonionic surfactant available.

Builder granule B2

The following components were placed in an Eirich RV02 mixer and mixed together and granulated into a powder:

1180g zeolite 4A,
260g light soda ash, and
570g Sokalan CP5.

The resulting powder was dried in a Aeromatic Strea-1 fluid bed at a temperature of 80°C. The resulting powder B2 had the following composition:

Ingredients	B2 (wt%)
Zeolite 4A (anh)	55.2
Sodiumcarbonate	12.2
Sokalan CP5	10.7
Moisture	21.9

Detergent powders 2, 3 (invention) and B (comparative)

Nonionic granules N3, N4 and NX were mixed in various proportions with builder granules B2 and/or other ingredients to provide fully formulated powdered detergent compositions having suitable levels of surfactant (20%) and builder for use in fabric washing.

It is apparent from the following table that the compositions of the present invention show significantly improved properties such as flow-rate and stability (as measured by measurements of the quantity of nonionic surfactant leaching out of the detergent composition under the test conditions).

Ingredients (wt%)	2	B	3
Nonionic granule N3	33.9		
Nonionic granule NX		70.8	
Nonionic granule N4			30.9
Builder granule B2	64.8		69.1
Dense sodium carbonate	1.3		
Light soda ash		21.1	
CP5 granule (ex BASF)		8.1	
BD [g/l]	727	805	722
DFR [ml/s]	107	74	108
Rosin Rammler mean diameter (μm)	479	506	539
Polyamide filter, 1 wk, mean [mg]	13	20	12
Polyamide filter, 3 wk, mean [mg]	17	33	16

Example 4

A nonionic-surfactant-containing granular composition N6 according to the present invention was manufactured in a two-step process according to the invention. The storage stability of this composition was compared to a similar granule N5 prepared as described previously for granules N2-N4. The two step process was carried out as follows:

Step 1

2.5 kg of SiO_2 (Sorbosil TC) and 4.6 kg of nonionic surfactant (Synperonic A7) were mixed in a Fukae FS30 mixer for 15 secs using an agitator speed of 200 rpm and a chopper speed of 3000 rpm. The powder was subsequently discharged and left standing until the temperature of the powder was below 30°C.

Step 2

The powder made in Step 1 was mixed with 1.5 kg of a mixture of nonionic surfactant (Synperonic A7), fatty acid (Pristerene 4916) (weight ratio Synperonic:Pristerene = 85:15) and sodium hydroxide solution (50% NaOH). Granulation time was 15 secs, using an agitator speed of 200 rpm and a chopper speed of 3000 rpm. The powder was discharged and left to cool.

The formulation and storage stability of N6 was compared to the stability of N5.

Composition [wt%]	N6	N5
SiO_2 (Sorbosil TC 15)	28.7	26.1
Nonionic surfactant (Synperonic A7)	67.5	64.7
Soap	2.8	7.8
Water etc.	1.0	1.4
Polyamide filter, 1 week storage, mean [mg]	45	72
Polyamide filter, 1 week, mean [mg/g nonionic in sample]	2.7	4.5

As can be seen the structurant (soap) level in N6 is clearly lower than in N5. Furthermore, the surfactant level in N6 is higher. Not withstanding those two facts the storage stability of N6 is better.

Examples 5 to 8

These Examples show how nonionic granules and anionic granules can be used in conjunction with a base powder of low surfactant content, and/or a builder granule, to prepare detergent powders of high bulk density and high surfactant content having excellent powder properties.

Base powder F1

The following powder was prepared by spray-drying in a countercurrent tower with a diameter of 2.5 m.

Ingredients (wt%)	F1
STP	72.7
Sodium silicate	8.1
NaLAS	5.5
Water	13.7

Anionic granules A1

Primary alcohol sulphate (PAS) granules were prepared using a dryer/granulator supplied by VRV SpA, according to the following process. PAS paste containing 70% neutralised coco PAS and 30% water was dried in a dryer/granulator supplied by VRV SpA, Italy, using the following conditions. The temperature of the material entering 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 the drying and cooling zones were 10 m^2 and $5m^2$ 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. At each step, the process conditions were stabilised for 15 minutes. The particles then passed to a cooling zone operated at a temperature of 30°C.

Anionic granules A2

Linear alkylbenzene sulphonate (LAS) granules were also produced in the same apparatus, by neutralising LAS acid with sodium carbonate. Furthermore, zeolite MAP was dosed as a layering agent and sodium sulphate was dosed as well. A 1.2 m^2 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 final two sections. Zeolite was added via this port in the final section. An electrically-powered oil heater provided the heating to the first two jacket sections. 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 m^3/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 to 100 kg/hr. A degree of neutralisation of alkyl benzene sulphonate of greater than 95 was achieved.

The granules A1 and A2 had the following compositions:

Ingredients (wt%)	A2	A1
NaPAS		90
NaLAS	81	
Zeolite 4A	10	
Sodium carbonate	5	
Water	2	5
Miscellaneous	3	5

Builder granules B3 and B4

The following dense builder granules were produced:

Dense STP granules B3: STP powder was continuously brought in a Schugi Flexomix granulator, while spraying on a 10% alkaline silicate solution. The exiting material was cooled in a fluid bed, resulting in a granular powder of bulk density 744 g/l having the following composition:

Ingredients (wt%)	B3 (wt%)
STP	85.0
sodium silicate	2.3
water	12.7

Dense zeolite granules B4:

Zeolite MAP was continuously fed into a Lödige CB30 granulator, together with 40% Sokalan CP5 solution and water. The CB30 was typically operated at a speed of 1500 rpm. The resulting powder was continuously dried in a Niro fluid bed, using an air temperature of 200°C. The resulting powder had a BD of 850 g/l and the following composition:

Ingredients (wt%)	B4 (wt%)
Zeolite MAP (anh)	62.2
Sokalan CP5	20.7
water	17.1

The granules described above, and nonionic granule N5 described previously, were mixed together in various combinations, and with other post-dosed ingredients, to produce full detergent powder formulations as shown in the following table.

Formulation [wt%]	5	6	7	8
Base powder F1			35.8	
Builder granule B3				38.2
Builder granule B4	29.0	34.0		
Anionic granule A1		12.9		
Anionic granule A2			8.6	13.8
Nonionic granule N5	30.9	9.7	6.2	7.8
Light soda ash			10.0	
Dense granular sodium sulphate			25.5	6.5
Nabion 15 granules[1]	3.0	5.5		16.8
SCMC powder	1.0	1.0	1.0	1.3
Antifoam/fluorescer granules[2]	4.0	5.1	2.1	2.3
Sodium perborate monohydrate			6.5	8.5
Sodium percarbonate	23.0	19.0		
TAED granules	6.5	5.5	2.0	2.5
Dequest 2047	1.0	0.5	0.4	0.5
Dense granular carbonate		5.0		
Enzymes, perfume, coloured speckles etc	1.6	1.8	1.9	1.9
Total surfactant	20.0	17.9	12.9	16.2
Total nonionic	20.0	6.3	4.0	5.0
BD [g/l]	739	707	638	766
DFR [ml/s]	124	130	96	114

[1] Trade Mark: sodium carbonate/29% wt sodium silicate cogranules, ex Rhône-Poulenc.

[2] Silicone/silica antifoam granules.

In all cases, insolubles are below 5% in the abovementioned solubility test. Example 7 shows how standard low active sodium polyphosphate or zeolite built compositions can be post-dosed with anionic and nonionic surfactant containing granular compositions to boost the active level, to give powder compositions with acceptable flow rates. Example 5 shows how a powder composition can be made entirely out of nonionic granules, builder containing granules and post-dosed ingredients, to give a product with a good flow rate.

Examples 6 and 8 show how builder containing granules, nonionic surfactant containing granular compositions and anionic surfactant containing granules can be mixed with post-dosed ingredients to provide fully formulated compositions with good flow rates.

Example 9

In this Example, the importance of the oil carrying capacity of the silica carrier in the nonionic granule is demonstrated.

The following silicas were used to produce nonionic granules in accordance with the invention:

Granule N7: Sorbosil TC15 ex Crosfield
Granule N8: Sipernat D17 ex Degussa
Granule N9 Sipernat 50 ex Degussa
Granule N10: Aerosil 380 ex Degussa
Granule N11: Zeosyl 200 ex Huber

All have an oil absorbing capacity of 1.0 ml/g or higher. As a comparative material zeolite MAP ex Crosfield was used which has an oil absorbing capacity below 1.0 ml/g.

Granules were prepared by dosing the silica powder into a Moulinex Multi Moulinette kitchen mixer. Into the Moulinex a mixture containing 85 wt% nonionic (Synperonic A7 ex ICI) and 15 wt% fatty acid (Pristerene 4916 ex Unichema) was dosed at a temperature of around 60°C. Furthermore a stoichiometric amount of 50% NaOH solution was dosed to neutralise the fatty acid. The mixture was granulated for 10 seconds, discharged and left to cool. In all cases powders with good granulometry were obtained.

The following nonionic surfactant levels were obtained:

Powder	Silica	Oil absorption of silica [ml/g]	Nonionic level in powder [wt%]
N7	Sorbosil TC15	2.8	64
N8	Sipernat D17	2.3	57
N9	Sipernat 50	3.3	66
N10	Aerosil 380	3.5	65
N11	Zeosyl 200	2.6	61
NY (Comparative)	Zeolite MAP	0.6	28

As can be seen from the comparative example, high absorption capacities are required to achieve the desired nonionic surfactant loadings.

Example 10

Two more nonionic granules N12 and N13 for use in detergent compositions according to the invention were produced, on a larger scale using a continuous granulation process.
N12 contained soap as a structurant, while N13 contained glucose.

The process route consisted of a Lödige CB30, followed by a Niro fluid 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 m^3/hr. The total flow of powder exiting the process was in the order of 600 kg/h.

Granule N12: Sorbosil TC15 was continuously dosed into the CB30, into which also a mixture of nonionic surfactant (Synperonic A7 ex ICI) and fatty acid (Pristerene 4916) was dosed via dosing pipes. At 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 bags. Coarse particles (>1400µm) were separated from the product by the Mogensen sieve.

Granule N13: Sorbosil TC15 was continously dosed into the CB30, into which also a nonionic surfactant (Synperonic A7 ex ICI) was dosed via dosing pipes. At the same time a 40% glucose solution was was dosed. 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 treated with air which had a temperature of 80-120°C. Fines were filtered from the air stream with a cyclone and filter bags. Coarse particles (>1400µm) were separated from the product by the Mogensen sieve.

The resulting granules had the formulations and properties shown in the table below.

Composition [wt%]	N12	N13
Sorbosil TC15	33.6	27.7
Synperonic A7	55.6	58
Soap	9.8	
Glucose		10.8
Water	1	3.5
BD [g/l]	570	607
DFR [ml/s]	143	129

Examples 11 and 12, Comparative Examples C and D

In these Examples the benefit of using a separate nonionic granule in a powder will be illustrated by comparing the physical properties of that powder with those of a similar powder to which nonionic is sprayed on.

A spray-dried detergent base powder F2 was prepared by making a slurry containing NaLAS, Synperonic A7, STP, silicate and water and drying the slurry in a countercurrent spray-drying tower to produce base powder F2 having the following composition:

Ingredients	F2 (wt%)
NaLAS	27.4
Nonionic	3.1
STP	43.6
Silicate	11.6
Moisture, salts etc	14.3

For Comparative Examples C and D, nonionic surfactant was sprayed onto powder F2 by dosing 1880 g of this powder into an Eirich RV02 mixer and adding 120 g of Synperonic A7 while the mixer was operated at 400 rpm.

For Examples 11 and 12, the granule N13 was used as the source of additional nonionic surfactant.

Postdosed ingredients were added and the final formulations were as shown in the table below.

Ingredients [wt%]	C	11	D	12
F2 + nonionic spray-on	95.7		47.9	
F2		90.0		45.0
Nonionic granule N13		10.0		5.1
Dense carbonate	4.3		12.2	10.0
Dense sulphate			12.5	12.5
Perborate tetrahydrate			23.0	23.0
TAED			2.8	2.8
Antifoam granule			1.0	1.0
Dequest 2047			0.6	0.6
PVP granule			0.1	0.1
Total surfactant	33.2	33.3	16.6	16.7
Total nonionic surfactant	8.5	8.6	4.3	4.3
BD [g/l]	356	386	555	584
DFR [ml/s]	38	107	91	121

As can be seen, the flow rates of the invention products are substantially higher than of the comparative examples indicating that the use of separate nonionic granules is beneficial.

Example 13

This Example illustrates the production of further nonionic granules containing a range of structurants using a two-step process.

Nonionic granules N14 to N19 were prepared via the two step process in a Moulinex Multi Moulinette kitchen mixer. Silica and nonionic surfactant were dosed in the Moulinex and mixed together for 10 seconds, after which the mixture was cooled to approximately 30°C. In the second step aqueous solutions of a structurant were added and the mixture was granulated for another 10 seconds. The resulting powder was dried in a Aeromatic Strea-1 fluid bed at 80°C.

The following structurants were used (wt% in water):

Granule N14:	60% maltose
Granule N15:	30.5% trisodium citrate + 5.2 % Sokolan CP5
Granule N16:	15% glucose + 8% Sokolan CP5
Granule N17:	19.5% sodium chloride + 10.4% Sokolan CP5
Granule N18:	15% polyvinylalcohol (Mowiol 4-8 ex Hoechst)
Granule N19:	18% sugar

Free-flowing granules with the following levels of nonionic surfactant and structurant were produced:

Powder	N14	N15	N16	N17	N18	N19
Structurant	maltose	citrate /CP5	glucose /CP5	NaCl /CP5	PVA	sugar
level [wt%]	12.4	12.4	9.2	6.0	4.5	4.6
Nonionic level [wt%]	55.0	56.3	58.9	60.1	61.1	61.2

Examples 14 to 17

These Examples illustrate formulations according to the invention containing very high surfactant levels.

Base powder F3 was prepared by making a slurry containing water, NaLAS, STP, silicate, sodium sulphate, SCMC and fluorescer. This slurry was spray-dried in a countercurrent spray-drying tower, resulting in the following composition:

Ingredients	F3 (wt%)
NaLAS	37.9
STP	23.3
Silicate	11.0
Sodium sulphate	17.3
Moisture, minors	10.5

Base powder F4 was prepared by using a Lödige CB30 mixer, in which the various ingredients were mixed together, followed by a densification step in a Lödige KM300 mixer. The resulting powders were cooled in a fluid bed. In the CB30 mixer, phosphate and sodium carbonate were dosed as solid components.

LAS acid was dosed and neutralised with the sodium carbonate to make NaLAS. At the same time a 40% Sokalan CP5 solution was dosed.

The CB30 was operated at 1500 rpm and the exiting powder was layered with zeolite MAP prior to entering the KM300. After cooling in the fluid bed, powder was collected with the following composition:

Ingredients [wt%]	Base powder F4
NaLAS	21.1
STP	54.3
Sodium carbonate	12.3
zeolite MAP (anh)	4.0
Sokalan CP5	2.6
Moisture, minors etc	5.7

Builder granule B5 was prepared by the process described above for builder granule B3 (see Examples 7 to 10).

Ingredients [wt%]	Builder granule B5
STP	89.3
Silicate	1.8
Moisture, minors etc	8.9

A soap-structured nonionic granule N20 similar to granule N12 (Example 10) was prepared by the same process.

Composition [wt%]	N20
Sorbosil TC15	30.0
Lutensol AO7	55.0
Soap	13.1
Water	1.9

Anionic granule A3 was prepared by the process described earlier for granule A2 (Examples 5 to 8), using a $2m^2$ VRV machine:

Composition [wt%]	A3
NaLAS	70
Zeolite 4A	20
Zeolite MAP	5
Moisture, NDOM etc	5

With these ingredients the following powders having very high surfactant contents and excellent flow properties were assembled:

Formulation	14	15	16	17
F3	60.2			
F4		46.1		
B5	12.2		28	
N20	27.6	27.6	27.6	80
A3		18.6	32.6	
Granular carbonate		7.7	11.8	20
Total surfactant [%]	38	38	38	44
BD [g/l]	353	675	746	646
DFR [ml/s]	122	129	139	137

CLAIMS

1 A particulate detergent composition characterised in that it is composed of at least two different granular components:

(a) a nonionic-surfactant-containing granular component characterised in that it comprises:

(a1) more than 55% by weight of nonionic surfactant,

(a2) at least 5% by weight of silica having an oil absorption capacity of at least 1.0 ml/g,

(a3) optionally, less than 10% by weight of aluminosilicate,

(b) at least one other granular component.

2 A detergent composition as claimed in claim 1, characterised in that it comprises at least one other granular component (b) selected from

(b1) a spray-dried or agglomerated base powder containing anionic surfactant, builder and, optionally, nonionic surfactant,

(b2) a builder granule, and

(b3) a granule containing at least 60% by weight of anionic surfactant.

3 A detergent composition as claimed in any preceding claim, characterised in that the nonionic-surfactant-containing granular component (a) contains at least 59% by weight of nonionic surfactant.

4 A detergent composition as claimed in any preceding claim, characterised in that the nonionic-surfactant-containing granular component (a) contains at least 10% by weight of silica.

5 A detergent composition as claimed in claim 4, characterised in that the nonionic-surfactant-containing granular component (a) contains at least 15% by weight of silica.

6 A detergent composition as claimed in any preceding claim, characterised in that the silica in the nonionic-surfactant-containing granular component (a) has an oil absorption capacity of at least 1.5 g/l.

7 A detergent composition as claimed in claim 6, characterised in that the silica in the nonionic-surfactant-containing granular component (a) has an oil absorption capacity of at least 2.0 g/l.

8 A detergent composition as claimed in any preceding claim, characterised in that the nonionic-surfactant-containing granular component (a) contains less than 5% by weight of aluminosilicate.

9 A detergent composition as claimed in any preceding claim, characterised in that the nonionic-surfactant-containing granular component (a) contains from 2 to 15% by weight of a structurant.

10 A detergent composition as claimed in claim 9, characterised in that the nonionic-surfactant-containing granular component (a) contains a structurant selected from soaps, sugars, succinates, silicates, citrates, polyethylene/propylene glycol of molecular weight 1000 to 12 000, polyacrylate of molecular weight 30 000 to 200 000, polyvinyl alcohol of molecular weight 30 000 to 200 000, acrylate/maleate copolymers, and mixtures thereof.

11 A detergent composition as claimed in claim 10, characterised in that the nonionic-surfactant-containing granular component (a) contains a structurant selected from polyethylene glycol, soap, maltose, glucose, sucrose, polyvinyl alcohol, and acrylate/maleate copolymer in admixture with glucose, sodium chloride or trisodium citrate.

12 A detergent composition as claimed in any preceding claim, characterised in that it comprises from 2 to 50% by weight of the nonionic-surfactant-containing granular component (a) and from 50 to 98% by weight of one or more other granular components (b), the percentages being based on the total amount of granular components (a) and (b).

13 A process for the preparation of a nonionic-surfactant-containing granular composition containing more than 55% by weight of nonionic surfactant, at least 5% by weight of silica having an oil absorption capacity of at least 1.0 ml/g, from 5 to 15% by weight of a structurant and optionally less than 10% by weight of aluminosilicate, characterised in that it comprises the steps of:

(i) mixing from 70 to 100% by weight of the solid components, from 70 to 100% by weight of the nonionic surfactant and less than 50% by weight of the structurant, and

(ii) adding the remainder of the nonionic surfactant and solid components and mixing further.

14 A process as claimed in claim 13, characterised in that at least 80% by weight, preferably at least 90% by weight, of the solid components are added in step (i).

15 A process as claimed in claim 13 or claim 14, characterised in that from 2 to 10% by weight of structurant is added to the components in the mixer in step (i).

16 A process as claimed in any one of claims 13 to 15, characterised in that at least 70% by weight of the structurant is added in step (ii).

17 A nonionic-surfactant-containing granular composition containing more than 55% by weight of nonionic surfactant, at least 5% by weight of silica having an oil absorption capacity of at least 1.0 ml/g, from 5 to 15% by weight of a structurant and optionally less than 10% by weight of aluminosilicate, prepared by a process as claimed in any one of claims 13 to 16.

18 A particulate detergent composition as claimed in claim 1, characterised in that the nonionic-surfactant-containing granular composition (a) is prepared by a process as claimed in any one of claims 13 to 16.

INTERNATIONAL SEARCH REPORT

International Application No
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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D3/12 C11D17/06 C11D17/00

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C11D

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.
X	DATABASE WPI Week 9614 Derwent Publications Ltd., London, GB; AN 96-136608 XP002077762 & JP 08 027498 A (KAO CORP.) , 30 January 1996 see abstract & JP 08 027498 A (...) cited in the application --- -/--	1,4,5, 10,13,17

☒ Further documents are listed in the continuation of box C.

☐ Patent family members are listed in annex.

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Date of the actual completion of the international search 16 September 1998	Date of mailing of the international search report 28/09/1998
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Van Bellingen, I

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INTERNATIONAL SEARCH REPORT

national Application No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Week 9724 Derwent Publications Ltd., London, GB; AN 97-267896 XP002077763 & JP 09 095694 A (LION CORP.) , 8 April 1997 see abstract -----	1,13,17

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