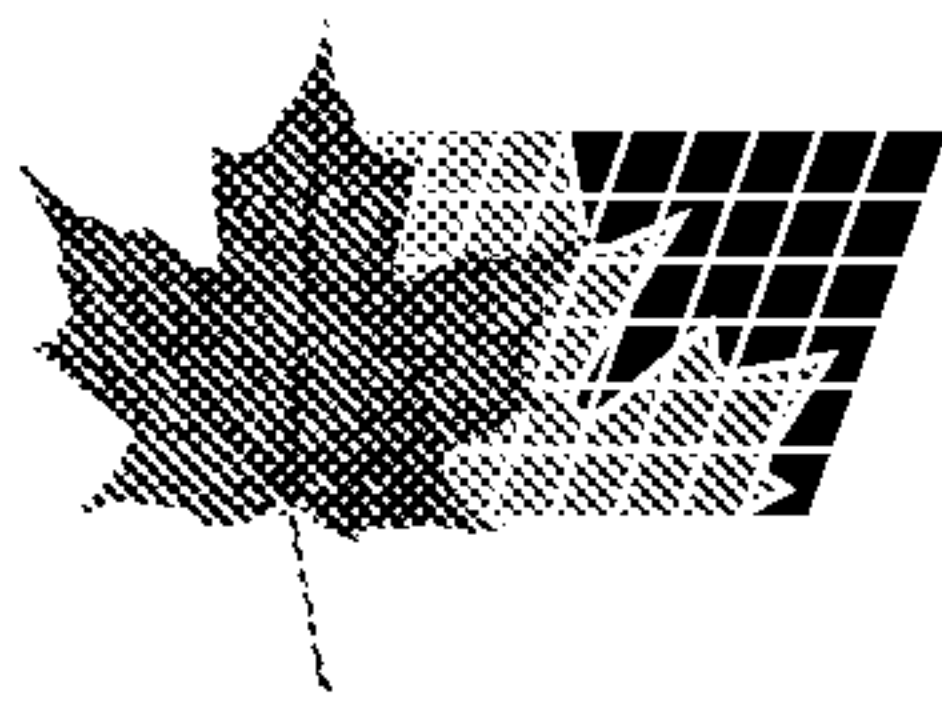




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(54) **PROCESSUS DE PRODUCTION DE GRANULES D'AGENT DE
SURFACE**

(54) **A PROCESS FOR THE PRODUCTION OF SURFACTANT
GRANULES**

(57) The invention relates to a process for the production of surfactant granules containing nonionic surfactants and other detergent ingredients in which the solid and optionally liquid ingredients are granulated in the presence of a mixture of nonionic surfactant and a nonaqueous solvent and which is characterized in that the nonaqueous solvent is polyalkylene glycol liquid at 80°C. Granules with a narrow particle spectrum and good solubility are obtained.

Abstract

The invention relates to a process for the production of surfactant granules containing nonionic surfactants and other detergent ingredients in which the solid and optionally liquid ingredients are granulated in the presence of a mixture of nonionic surfactant and a nonaqueous solvent and which is characterized in that the nonaqueous solvent is polyalkylene glycol liquid at 80°C. Granules with a narrow particle spectrum and good solubility are obtained.

A Process for the Production of Surfactant Granules

Field of the Invention

This invention relates to a process for the production of surfactant granules containing nonionic surfactants in which the solid and optionally liquid constituents are granulated in the presence of a mixture of nonionic surfactant and a nonaqueous solvent.

5

Background of the Invention

Compacted or highly concentrated detergent powders or tablets represent a substantial percentage of commercially available detergents. Such detergents are generally not obtained by a spray drying process, but
10 rather by mixing, granulation and compacting processes in which high temperatures are avoided. On account of the relatively low temperatures, the removal of water introduced by the starting materials is relatively laborious. In many cases, a drying step has to be carried out on completion of the granulation process.

15 Another problem lies in the poor solubility of the relatively heavily compacted particles. This problem is often encountered in particular with formulations containing a high percentage of alkoxylated fatty alcohols. An improvement in solubility can be achieved by incorporating alkyl polyglycosides in the granules. However, the incorporation of the alkyl polyglycosides is problematical because they cannot be processed by spray
20 drying processes and the resulting particles are extremely tacky.

The removal of water from the granules is often incomplete and leads to products with a variable water content. If the granules are to be further processed to tablets which are expected to show rapid and constant
25 solubility in water, the use of granules with a variable water content can lead to tablets with variable dissolving rates.

The production of surfactant granules for use in detergents is known from the prior art. Thus, International patent application **WO 97/09415**

discloses a process for the production of a non-spray-dried particulate surfactant composition which has a bulk density of at least 600 g/l and which contains a polymeric builder component and/or a soil-release polymer. The granulation liquid and solid constituents are mixed in a granulator and the polymer is added in admixture with a non-aqueous diluent during the granulation step. An ethoxylated nonionic surfactant is preferably used as the non-aqueous diluent.

International patent application **WO 97/02338** discloses a process for the production of particulate surfactant compositions with a bulk density below 700 g/l. To produce the surfactant composition disclosed in this document, a particulate starting material which contains at least 10% by weight of a component with a bulk density of not more than 600 g/l and which is not a surfactant compound is mixed with a liquid binder in a granulator and then granulated. The particulate starting material contains a builder. Surfactant or a surfactant precursor is present in the particulate starting material and/or in the binder.

International patent application **WO 97/22685** discloses a process for the production of particulate detergent compositions with bulk densities of 300 to 1300 g/l. In the process disclosed in this document, particulate starting material is mixed with a liquid binder and partly granulated. The partly granulated mixture is transferred to a less shear-intensive granulator, more liquid binder is added and the granulation process is continued until a particulate powder with the required bulk density is obtained. Water, anionic surfactant, nonionic surfactant or mixtures thereof may be used as the liquid binder.

European patent application **0 799 884** describes a process for the production of free-flowing particulate surfactant compositions containing alkyl polyglycosides in which a concluding drying step is said to be unnecessary. To carry out the described process, a mixture of a water-containing alkyl polyglycoside paste, an ethoxylated nonionic surfactant

and a solid water-soluble inorganic salt is initially prepared. The mixture obtained separates into an organic phase and a water-rich phase, the organic phase containing alkyl polyglycoside, ethoxylated nonionic surfactant and water being separated off. The percentages of the individual components in the first step is selected so that the ratio of alkyl polyglycoside to ethoxylated nonionic surfactant in the organic phase is in the range from 35:65 to 65:35 and the ratio of ethoxylated nonionic surfactant to the total quantity of water is in the range from 90:10 to 60:10. To produce the surfactant compositions, the mixture obtained and, optionally, other surfactants are mixed with one or more particulate carrier materials and the resulting mixture is processed in a conventional high-speed mixer or granulator to form a particulate product.

The processes described above have the disadvantage that they are either very complicated to carry out or lead to non-free-flowing products with - in some cases - poor solubility.

Another disadvantage is that the products obtained have a broad particle size distribution. Where the granules produced are further processed to products of larger particle size, detergent tablets, uniform products are only obtained when the starting components used show little variation in their properties.

The problem addressed by the present invention was to provide a process for the production of surfactant granules which would not be attended by the disadvantages mentioned above. More particularly, the present invention set out to provide a process which would give granules characterized by good and constant solubility and by a narrow particle spectrum. In addition, the granules obtained would show such favorable free flow behavior that there would be no need for an additional drying step.

Description of the Invention

It has now surprisingly been found that the disadvantages of the

prior art can be remedied by granulating the solid and liquid detergent ingredients in the presence of a mixture of nonionic liquid surfactant and a polyethylene glycol liquid at 80°C as nonaqueous solvent. Free-flowing granules with good solubility and a narrow particle spectrum are obtained.

5 Accordingly, the present invention relates to a process for the production of surfactant granules containing nonionic surfactants and other detergent ingredients in which the solid and optionally liquid ingredients are granulated in the presence of a mixture of nonionic surfactant and a nonaqueous solvent, characterized in that the nonaqueous solvent is a
10 polyalkylene glycol liquid at 80°C.

The polyalkylene glycol used in accordance with the invention may also be a mixture of several alkylene glycols. A preferred polyalkylene glycol is polyethylene glycol, more particularly a polyethylene glycol with a molecular weight of 100 to 10,000 and more particularly in the range from
15 300 to 4,000.

Suitable nonionic surfactants are any liquid nonionic surfactants suitable for use in detergents. Preferred nonionic surfactants are alkoxylated, advantageously ethoxylated, more especially primary alcohols preferably containing 8 to 18 carbon atoms and, on average, 1 to 12 moles
20 of ethylene oxide (EO) per mole of alcohol, in which the alcohol group may be linear or, preferably, methyl-branched in the 2-position or may contain linear and methyl-branched groups in the form of the mixtures typically present in oxoalcohol groups. However, alcohol ethoxylates containing linear groups of alcohols of native origin with 12 to 18 carbon atoms, for
25 example coconut, palm, tallow or oleyl alcohol, and on average 2 to 8 EO per mole of alcohol are particularly preferred. Preferred ethoxylated alcohols include, for example, C₁₂₋₁₄ alcohols containing 3 EO or 4 EO, C₉₋₁₁ alcohols containing 7 EO, C₁₃₋₁₅ alcohols containing 3 EO, 5 EO, 7 EO or 8 EO, C₁₂₋₁₈ alcohols containing 3 EO, 5 EO or 7 EO and mixtures thereof,
30 such as mixtures of C₁₂₋₁₄ alcohol containing 3 EO and C₁₂₋₁₈ alcohol

containing 7 EO. The degrees of ethoxylation mentioned represent statistical mean values which, for a special product, can be a whole number or a broken number. Preferred alcohol ethoxylates have a narrow homolog distribution (narrow range ethoxylates, NRE).

5 Besides the fatty alcohol alkoxylates preferably used, other nonionic surfactants, such as alkyl polyglycosides, alkoxylated, preferably ethoxylated or ethoxylated and propoxylated C₈₋₁₈ fatty acid alkyl esters, N-fatty alkyl amine oxides, polyhydroxyfatty acid amides and mixtures thereof may also be used.

10 The nonionic surfactant content of the final granules is preferably between 5% by weight and 25% by weight.

Particularly good results are obtained in the process according to the invention if the nonionic surfactant and the polyalkylene glycol are used in a mixing ratio of 10:1 to 1:10.

15 Inorganic and organic builders and other surfactants, more particularly anionic surfactants, may be incorporated in the surfactant granules as additional constituents. The detergents according to the invention may additionally contain any substances typically present in detergents, such as inorganic salts, bleaching agents, bleach activators,
20 redeposition inhibitors, foam inhibitors, salts of polyphosphonic acids, optical brighteners, enzymes and mixtures thereof.

In one preferred embodiment, the other components are incorporated in the form of a spray-dried powder in the second step of the process. Temperature-sensitive or water-sensitive components may be separately
25 added.

Suitable builders are any builders suitable for detergents which have a sufficiently large inner surface to be able to absorb the nonionic surfactant. Examples of inorganic builders are zeolites, crystalline layer silicates, amorphous silicates, soda, phosphates and mixtures thereof,
30 zeolite being preferred.

The zeolite used may be, for example, finely crystalline synthetic zeolite containing bound water, such as zeolite A, zeolite P and mixtures of A and P. An example of a commercially available zeolite P is zeolite MAP® (a Crosfield product).

5 Zeolites of the faujasite type are mentioned as other preferred and particularly suitable zeolites. Together with zeolites X and Y, the mineral faujasite belongs to the faujasite types within zeolite structure group 4 which is characterized by the double 6-membered ring subunit D6R (cf. Donald W. Breck: **"Zeolite Molecular Sieves"**, John Wiley & Sons, New
10 York, London, Sydney, Toronto, 1974, page 92). Besides the faujasite types mentioned, the minerals chabasite and gmelinite and the synthetic zeolites R (chabasite type), S (gmelinite type), L and ZK-5 belong to zeolite structure group 4. The last two of these synthetic zeolites do not have any mineral analogs.

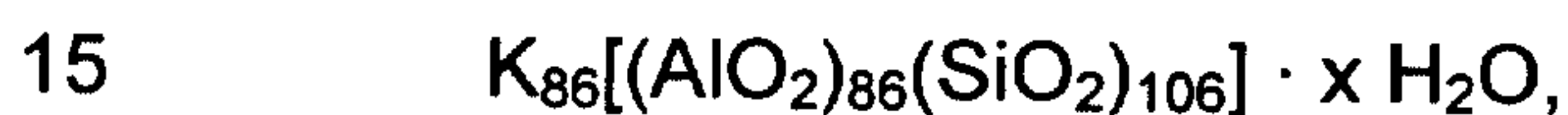
15 Faujasite zeolites are made up of β -cages tetrahedrally linked by D6R subunits, the β -cages being arranged similarly to the carbon atoms in diamond. The three-dimensional framework of the faujasite zeolites used in the process according to the invention has pores 2.2 and 7.4 Å in size. In addition, the elementary cell contains eight cavities each ca. 13 Å in
20 diameter and may be described by the formula $\text{Na}_{86}[(\text{AlO}_2)_{86}(\text{SiO}_2)_{106}] 264 \text{H}_2\text{O}$. The framework of the zeolite X contains a void volume of around 50%, based on the dehydrated crystal, which represents the largest empty space of all known zeolites (zeolite Y: ca. 48% void volume, faujasite: ca. 47% void volume). (All data from: Donald W. Breck: **"Zeolite Molecular
25 Sieves"**, John Wiley & Sons, New York, London, Sydney, Toronto, 1974, pages 145, 176, 177).

In the context of the present invention, the expression "faujasite zeolite" characterizes all three zeolites which form the faujasite subgroup of zeolite structure group 4. According to the invention, therefore, zeolite Y
30 and faujasite and mixtures of these compounds may also be used in

addition to zeolite X although pure zeolite X is preferred. Mixtures or co-crystallizates of faujasite zeolites with other zeolites, which do not necessarily have to belong to zeolite structure group 4, may also be used in accordance with the invention, the advantages of the process according to the invention coming to light particularly clearly when at least 50% of the zeolites are faujasite zeolites.

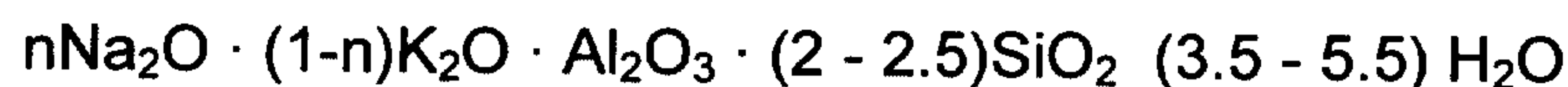
The aluminium silicates used in the process according to the invention are commercially obtainable and the methods for their production are described in standard works.

Examples of commercially available X-type zeolites may be described by the following formulae:



in which x may assume a value of 0 to 276 and which have pore sizes of 8.0 to 8.4 Å.

For example, a co-crystallizate of zeolite X and zeolite A (ca. 80% by weight zeolite X), which is marketed by CONDEA Augusta S.p.A. under the name of VEGOBOND AX[®] and which may be described by the following formula:



is commercially obtainable and may be used with advantage in the process according to the invention.

Zeolites of the Y type are also commercially obtainable and may be described, for example, by the following formulae:



5



in which x is a number of 0 to 276 and which have pore sizes of 8.0 Å.

10 The particle sizes of the faujasite zeolites used in the process according to the invention is in the range from 0.1 to 100 µm, preferably in the range from 0.5 to 50 µm and more preferably in the range from 1 to 30 µm, as measured by standard methods for determining particle size.

Other suitable builders are, for example, crystalline layer-form sodium silicates corresponding to the general formula $\text{NaMSi}_x\text{O}_{2x+1}\text{A} \cdot y \text{H}_2\text{O}$, where M is sodium or hydrogen, x is a number of 1.9 to 4 and y is a number of 0 to 20, preferred values for x being 2, 3 or 4. Crystalline layer silicates such as these are described, for example, in European patent application **EP-A-0 164 514**. Preferred crystalline layer silicates corresponding to the above formula are those in which M is sodium and x assumes the value 2 or 3. Both β- and δ-sodium disilicates $\text{Na}_2\text{Si}_2\text{O}_5\text{A} \cdot y \text{H}_2\text{O}$ are particularly preferred, β-sodium disilicate being obtainable, for example, by the process described in International patent application **WO-A-91/08171**.

25 Other suitable builders are amorphous sodium silicates with an $\text{Na}_2\text{O}:\text{SiO}_2$ ratio (modulus) of 1:2 to 1:3.3, preferably 1:2 to 1:2.8 and more preferably 1:2 to 1:2.6 which preferably dissolve with delay and have multiple wash cycle properties. The delay in dissolution in relation to conventional amorphous sodium silicates can have been obtained in various ways, for example by surface treatment, compounding, compacting
30 or by overdrying. In the context of the invention, the term amorphous is

also understood to encompass "X-ray amorphous". In other words, the silicates do not produce any of the sharp X-ray reflexes typical of crystalline substances in X-ray diffraction experiments, but at best one or more maxima of the scattered X-radiation which have a width of several degrees of the diffraction angle. However, particularly good builder properties may even be achieved where the silicate particles produce crooked or even sharp diffraction maxima in electron diffraction experiments. This may be interpreted to mean that the products have microcrystalline regions between 10 and a few hundred nm in size, values of up to at most 50 nm and, more particularly, up to at most 20 nm being preferred. So-called X-ray amorphous silicates such as these, which also dissolve with delay in relation to conventional waterglasses, are described for example in German patent application **DE-A-44 00 024**. Compacted amorphous silicates, compounded amorphous silicates and overdried X-ray-amorphous silicates are particularly preferred.

Useful organic carrier materials are, for example, the polycarboxylic acids usable, for example, in the form of their sodium salts, polycarboxylic acids in this context being understood to be carboxylic acids which bear more than one acid function. Examples of such carboxylic acids are citric acid, adipic acid, succinic acid, glutaric acid, malic acid, tartaric acid, maleic acid, fumaric acid, sugar acids, aminocarboxylic acids, nitrilotriacetic acid (NTA), providing their use is not ecologically unsafe, and mixtures thereof. Preferred salts are the salts of the polycarboxylic acids, such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids and mixtures thereof.

The acids per se may also be used. Besides their builder effect, the acids also typically have the property of an acidifying component and, hence, also serve to establish a relatively low and mild pH value in detergents. Citric acid, succinic acid, glutaric acid, adipic acid, gluconic acid and mixtures thereof are particularly mentioned in this regard.

Other suitable builders are polymeric polycarboxylates such as, for example, the alkali metal salts of polyacrylic or polymethacrylic acid, for example those with a relative molecular weight of 500 to 70,000 g/mole.

The molecular weights mentioned in this specification for polymeric polycarboxylates are weight-average molecular weights M_w of the particular acid form which, basically, were determined by gel permeation chromatography (GPC) using a UV detector. The measurement was carried out against an external polyacrylic acid standard which provides realistic molecular weight values by virtue of its structural similarity to the polymers investigated. These values differ distinctly from the molecular weights measured against polystyrene sulfonic acids as standard. The molecular weights measured against polystyrene sulfonic acids are generally higher than the molecular weights mentioned in this specification.

Particularly suitable polymers are polyacrylates which preferably have a molecular weight of 2,000 to 20,000 g/mole. By virtue of their superior solubility, preferred representatives of this group are the short-chain polyacrylates which have molecular weights of 2,000 to 10,000 g/mole and, more particularly, 3,000 to 5,000 g/mole.

Also suitable are copolymeric polycarboxylates, particularly those of acrylic acid with methacrylic acid and those of acrylic acid or methacrylic acid with maleic acid. Acrylic acid/maleic acid copolymers containing 50 to 90% by weight of acrylic acid and 50 to 10% by weight of maleic acid have proved to be particularly suitable. Their relative molecular weights, based on the free acids, are generally in the range from 2,000 to 70,000 g/mole, preferably in the range from 20,000 to 50,000 g/mole and more preferably in the range from 30,000 to 40,000 g/mole.

The (co)polymeric polycarboxylates may be used either in powder form or in the form of an aqueous solution. The content of (co)polymeric polycarboxylates in the detergent is preferably from 0.5 to 20% by weight and more preferably from 3 to 10% by weight.

In order to improve solubility in water, the polymers may also contain allyl sulfonic acids, such as allyloxybenzene sulfonic acid and methallyl sulfonic acid (cf. for example **EP-B-727 448**), as monomer.

Other particularly preferred polymers are biodegradable polymers of
5 more than two different monomer units, for example those which contain salts of acrylic acid and maleic acid and vinyl alcohol or vinyl alcohol derivatives as monomers in accordance with **DE-A-43 00 772** or those which contain salts of acrylic acid and 2-alkylallyl sulfonic acid and sugar derivatives as monomers in accordance with **DE-C-42 21 381**.

10 Other preferred copolymers are those which are described in German patent applications **DE-A-43 03 320** and **DE-A-44 17 734** and which preferably contain acrolein and acrylic acid/acrylic acid salts or acrolein and vinyl acetate as monomers.

Other suitable organic builders are dextrans, for example oligomers
15 or polymers of carbohydrates which may be obtained by partial hydrolysis of starches. The hydrolysis may be carried out by standard methods, for example acid- or enzyme-catalyzed methods. The end products are preferably hydrolysis products with average molecular weights of 400 to 500,000 g/mole. A polysaccharide with a dextrose equivalent (DE) of 0.5 to
20 40 and, more particularly, 2 to 30 is preferred, the DE being an accepted measure of the reducing effect of a polysaccharide by comparison with dextrose which has a DE of 100. Both maltodextrins with a DE of 3 to 20 and dry glucose sirups with a DE of 20 to 37 and also so-called yellow dextrans and white dextrans with relatively high molecular weights of 2,000
25 to 30,000 g/mole may be used. A preferred dextrin is described in British patent application **94 19 091**.

The oxidized derivatives of such dextrans are their reaction products with oxidizing agents which are capable of oxidizing at least one alcohol function of the saccharide ring to the carboxylic acid function. Dextrans thus
30 oxidized and processes for their production are known, for example, from

European patent applications **EP-A-0 232 202**, **EP-A-0 427 349**, **EP-A-0 472 042** and **EP-A-0 542 496** and from International patent applications **WO 92/18542**, **WO 93/08251**, **WO 93/16110**, **WO 94/28030**, **WO 95/07303**, **WO 95/12619** and **WO 95/20608**. An oxidized oligosaccharide
5 corresponding to German patent application **DE-A-196 00 018** is also suitable. A product oxidized at C₆ of the saccharide ring can be particularly advantageous.

Other suitable co-builders are oxydisuccinates and other derivatives of disuccinates, preferably ethylenediamine disuccinate. Ethylenediamine-
10 N,N'-disuccinate (EDDS), of which the synthesis is described for example in **US 3,158,615**, is preferably used in the form of its sodium or magnesium salts. The glycerol disuccinates and glycerol trisuccinates described, for example, in **US 4,524,009** and **US 4,639,325**, in European patent application **EP-A-0 150 930** and in Japanese patent application **JP**
15 **93/339896** are also preferred in this connection. The quantities used in zeolite-containing and/or silicate-containing formulations are from 3 to 15% by weight.

Other useful organic co-builders are, for example, acetylated hydroxycarboxylic acids and salts thereof which may optionally be present
20 in lactone form and which contain at least 4 carbon atoms, at least one hydroxy group and at most two acid groups. Co-builders such as these are described, for example, in International patent application **WO 95/20029**.

Other suitable carrier materials are oxidation products of polyglucosans containing carboxyl groups and/or water-soluble salts
25 thereof which are described, for example, in International patent application **WO-A-93/08251** or which the production is described, for example, in International patent application **WO-A-93/16110**. Oxidized oligosaccharides according to German patent application **DE 196 00 018** are also suitable.

Other preferred builders are polymeric aminodicarboxylic acids, salts or precursors thereof. Particular preference is attributed to polyaspartic acids or salts and derivatives thereof which, according to German patent application **DE-A-195 40 086**, are also said to have a bleach-stabilizing effect in addition to their co-builder properties.

Other suitable builders are polyacetals which may be obtained by reaction of dialdehydes with polyol carboxylic acids containing 5 to 7 carbon atoms and at least 3 hydroxyl groups, for example as described in European patent application **EP-A-0 280 223**. Preferred polyacetals are obtained from dialdehydes, such as glyoxal, glutaraldehyde, terephthalaldehyde and mixtures thereof and from polyol carboxylic acids, such as gluconic acid and/or glucoheptonic acid.

Another class of substances with co-builder properties are the phosphonates, more particularly hydroxyalkane and aminoalkane phosphonates. Among the hydroxyalkane phosphonates, 1-hydroxyethane-1,1-diphosphonate (HEDP) is particularly important as a co-builder. It is preferably used in the form of the sodium salt, the disodium salt showing a neutral reaction and the tetrasodium salt an alkaline reaction (pH 9). Preferred aminoalkane phosphonates are ethylenediamine tetramethylene phosphonate (EDTMP), diethylenetriamine pentamethylenephosphonate (DTPMP) and higher homologs thereof. They are preferably used in the form of the neutrally reacting sodium salts, for example as the hexasodium salt of EDTMP or as the hepta- and octasodium salts of DTPMP. Of the phosphonates, HEDP is preferably used as a builder. In addition, the aminoalkane phosphonates have a pronounced heavy metal binding capacity. Accordingly, it can be of advantage, particularly where the detergents also contain bleach, to use aminoalkane phosphonates, more particularly DTPMP, or mixtures of the phosphonates mentioned.

In addition, any compounds capable of forming complexes with alkaline earth metal ions may be used as co-builders.

Besides the nonionic surfactants used in accordance with the invention, the detergents according to the invention may also contain anionic surfactants such as, for example, C₈₋₂₂ alkyl sulfates, C₈₋₂₂ alkanesulfonates, C₈₋₂₂ olefin sulfonates, C₈₋₂₂ alkyl benzenesulfonates, C₈₋₂₂ fatty acid ether sulfates, C₈₋₂₂ fatty acid ester sulfonates, sulfonated fatty acid glycerol esters, 2,3-C₈₋₂₂-alkyl sulfates, salts, monoesters and/or diesters of alkylsulfosuccinic acid (sulfosuccinates), sulfuric acid monoesters of linear or branched C₇₋₂₁ alcohols ethoxylated with 1 to 6 moles ethylene oxide, fatty acid soaps and mixtures thereof.

The detergents according to the invention may additionally contain any substances typically present in detergents, such as inorganic salts, bleaching agents, bleach activators, redeposition inhibitors, foam inhibitors, salts of polyphosphonic acids, optical brighteners, enzymes and mixtures thereof.

In one preferred embodiment, the nonionic surfactant is first mixed with the polyalkylene glycol. The mixture obtained is applied to the builder in known manner. Normally, the builder is impregnated with the mixture obtained. In another process step, the impregnated builder is mixed with other constituents and granulated.

The granulation process may optionally be carried out using added auxiliaries, such as organic builders, for example polycarboxylic acids, citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids, aminocarboxylic acids, nitrilotriacetic acid (NTA), polymeric carboxylates, the sodium salts of the carboxylic acids mentioned above and mixtures thereof, providing these auxiliaries do not affect the solubility of the detergents according to the invention.

The granulation process may be carried out in a number of machines typically used in the detergent industry. For example, the spheronizers commonly used in the pharmaceutical industry may be used.

In machines such as these, the residence time of the granules is normally

less than 20 seconds. Conventional mixers and mixer/granulators may also be used for the granulation process. Suitable mixers are both high-shear mixers and also normal mixers operating at relatively low rotational speeds. Suitable mixers are, for example, Series R or RV Eirich® mixers (trademarks of Maschinenfabrik Gustav Eirich, Hardheim), the Schugi® Flexomix, the Fukae® FS-G mixers (trademarks of Fukae Powtech, Kogyo Co., Japan), Lödige® FM, KM and CB mixers (trademarks of Lödige Maschinenbau GmbH, Paderborn) and Series T or K-T Drais® mixers (trademarks of Drais-Werke GmbH, Mannheim). In all these mixers and spheronizers, the particles are converted into granules by liquid bridge binding of the non-aqueous binders. The residence times of the granules in the mixers is less than 60 seconds, the residence time also depending on the rotational speed of the mixer. The residence times are shorter, the faster the rotational speed of the mixer. The residence times of the granules in mixers/spheronizers are preferably less than 1 minute and more preferably under 15 seconds. In low-speed mixers, for example Lödige KM mixers, the residence times are up to 20 minutes, residence times of less than 10 minutes being preferred in the interests of process economy.

In the press agglomeration process, the surfactant-containing granules are compacted under pressure and under the effect of shear forces and, at the same time, homogenized and are then discharged from the machines via a shaping/forming stage. Industrially the most important press agglomeration processes are extrusion, roller compacting, pelleting and tableting. Press agglomeration processes preferably used in accordance with the present invention for producing the surfactant-containing granules are extrusion, roller compacting and pelleting.

The product obtained on completion of the granulation process is dry and does not have to be subjected to another drying step.

In order further to improve their processability and "dosability", the granules obtained may be powdered with an oil absorption component. As a result of this concluding powdering step using a fine-particle component, the liquids are fixed to the surface of the granules so that the granules are
5 unable to form lumps in storage. The oil absorption component should have an oil absorption capacity of at least 20 g/100 g, preferably of at least 50 g/100 g, more preferably of at least 80 g/100 g, more preferably of at least 120 g/100 g and, in one particular embodiment, of at least 140 g/100 g.

10 The oil absorption capacity is a physical property of a substance which can be measured by standardized methods. For example British Standards **BS1795** and **BS3483:Part B7:1982**, which both refer to **ISO 787/5**, are available. In these test methods, a weighed sample of the particular substance is applied to a dish and refined linseed oil (density:
15 0.93 gcm³) is added dropwise from a burette. After each addition, the powder is intensively mixed with the oil using a spatula, the addition of oil being continued until a paste of flexible consistency is obtained. This paste should flow without crumbling. Now, the oil absorption capacity is the quantity of oil added dropwise, based on 100 g of absorbent, and is
20 expressed in ml/100 g or g/100 g, conversions via the density of the linseed oil readily being possible.

The oil absorption component preferably has a small mean particle size because the active surface increases with decreasing particle size. Preferred detergent tablets contain a component with an oil absorption
25 capacity of at least 20 g/100 g which has a mean particle size below 50 µm, preferably below 20 µm and more preferably below 10 µm.

The oil absorption component may be selected from a number of substances. There are many inorganic and organic substances which have a sufficiently large oil absorption capacity. Fine-particle materials obtained
30 by precipitation are mentioned by way of example in this regard. Suitable

oil absorption components are, for example, silicates, aluminosilicates, calcium silicates, magnesium silicates and calcium carbonate. However, kieselgur (diatomaceous earth) and fine-particle cellulose fibers or derivatives thereof may also be used in accordance with the invention.

5 Preferred detergent tablets are characterized in that the component present in them with an oil absorption capacity of at least 20 g/100 g is selected from silicates and/or aluminosilicates, more particularly from the group of silicas and/or zeolites. These include, for example, fine-particle zeolites and also pyrogenic silicas (Aerosil®) or precipitated silicas.

10 The granules produced in accordance with the invention may either be used as sole detergent component or may be mixed and compounded with other particles containing other detergent ingredients.

In one preferred embodiment, the granules are mixed with other detergent ingredients and the resulting mixture is tableted to form
15 detergent tablets.

Embodiments of the invention are described in the following specific examples which are not to be construed as limiting.

Examples

20 To produce the surfactant granules, a spray-dried powder with the composition shown in Table 1, C₁₂₋₁₈ alkyl sulfate and sodium citrate were introduced into a 130 l Lödige mixer in the quantities shown in Table 2. A mixture of C_{12/18} fatty alcohol x 7 EO (Dehydol® LT 7) and polyethylene glycol with a molecular weight of 400 was then added and the mixture as a
25 whole was granulated. On completion of the granulation process, the granules were powdered with 3% Wessalith® XD (zeolite, a product of Degussa AG, Hanau).

In Comparison Example 1, a mixture of nonionic surfactant and glycerol was used for granulation; in Comparison Example 2, only nonionic
30 surfactant was used. The granules obtained were sieved to a particle size

of 0.6 to 1.6 mm. The granules with a particle size of 0.6 to 1.6 mm are referred to in Table 3 as "accepts".

The solubility of the products was tested by the so-called L Test. To this end, 8 g of substance were added to 1000 ml of water with a hardness of 16°dH at 30°C and stirred with a propeller stirrer for 1.5 minutes at 800 r.p.m. The undissolved solids were sieved on a 0.2 mm mesh sieve. The residue was dried to constant weight and weighed. The test results are set out in Table 4.

Table 1:

Composition of the tower powder in %

C ₁₂₋₁₈ alkyl benzenesulfonate	22.8
Tallow alcohol · 5 EO	1.3
C ₁₂₋₁₈ fatty acid soap	1.3
Sodium sulfate	4
Zeolite A	48
Phosphonate	1
Sokalan® CP5 [†]	8
NaOH	0.5
Water, salts	Balance

[†] Acrylic acid/maleic acid copolymer, product of BASF AG, Ludwigshafen

Table 2:

Granulation mixtures in % by weight, based on 100% of the components used

	E1	C1	C2
Tower powder	73	73	73
Sulfopon® 1218G ²	6	6	6
Na citrate	6	6	6
Dehydol® LT 7 ³	9.2	9.2	12
APG compound	2.8	/	/
Glycerol, 80%	/	2.8	/
Wessalith XD	3	3	3

² Compound containing 92% C₁₂₋₁₈ fatty alcohol sulfate (a product of Henkel KGaA)

³ C_{12/18} fatty alcohol · 7 EO (a product of Henkel KGaA)

Table 3:

Data of the granules

	E1	C1	C2
Fraction > 1.6 in %	10	6	8
Fraction < 0.6 in %	13	49	42
"Accepts" in %	77	45	50
Bulk density in g/l	650	610	660
L Test 1.5 mins.	5	10	16

The results set out in Table 3 show that the percentage of accepts in the granules produced in accordance with the invention is considerably higher than in the comparison granules and that the solubility of the granules produced in accordance with the invention is also distinctly better than that of the comparison granules.

CLAIMS

1. A process for the production of surfactant granules containing nonionic surfactants and other detergent ingredients in which the solid and optionally liquid ingredients are granulated in the presence of a mixture of
5 nonionic surfactant and a nonaqueous solvent, wherein the nonaqueous solvent is polyalkylene glycol liquid at 80°C.
2. A process as claimed in claim 1, wherein the polyalkylene glycol is a polyethylene glycol.
3. A process as claimed in claim 2, wherein the polyethylene glycol has
10 a molecular weight of 100 to 10,000.
4. A process as claimed in claim 3, wherein the polyethylene glycol has a molecular weight of 300 to 4,000.
5. A process as claimed in any one of claims 1 to 4, wherein alkoxyated primary alcohols containing 8 to 18 carbon atoms and on
15 average 1 to 12 moles alkylene oxide per mole alcohol are present as nonionic surfactants.
6. A process as claimed in any one of claims 1 to 5, wherein the nonionic surfactant and the polyalkylene glycol are used in a mixing ratio of 10:1 to 1:10.
- 20 7. A process as claimed in any one of claims 1 to 6, wherein inorganic and organic builders, other surfactants, inorganic salts, bleaching agents, bleach activators, redeposition inhibitors, foam inhibitors, salts of polyphosphonic acids, optical brighteners, enzymes or mixtures thereof are present as additional detergent ingredients.
- 25 8. A process as claimed in claim 7, wherein the other surfactants are anionic surfactants.
9. A process as claimed in claim 7 or 8, wherein the other detergent ingredients are used at least partly in the form of a spray-dried powder.

10. A process as claimed in any one of claims 1 to 9, wherein zeolites, crystalline layer silicates, amorphous silicates, soda and phosphates and mixtures thereof are used as builders.
- 5 11. A process as claimed in claim 10, wherein the zeolite contains at least 50% by weight of a faujasite zeolite.
12. A process as claimed in any one of claims 1 to 11, wherein the granules obtained are powdered with a component which has an oil absorption capacity of ≥ 20 g/100 g.
- 10 13. A process as claimed in any one of claims 1 to 12, wherein the granules obtained are not dried.
14. The use of the surfactant granules produced by the process claimed in any one of claims 1 to 13 for the production of detergent tablets.