



- (72) BOECKER, MONIKA, DE
(72) LIETZMANN, ANDREAS, DE
(72) KRUSE, HANS-FRIEDRICH, DE
(72) FEIST, MICHAEL, DE
(72) SCHAMBIL, FRED, DE
(72) BLASEY, GERHARD, DE
(72) JEBENS, HEINKE, DE
(71) HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN, DE
(51) Int.Cl.⁷ C11D 17/02, C11D 1/83, C11D 1/72, C11D 1/22, C11D 1/14
(54) **DETERGENT A LESSIVE ET PRODUIT DE NETTOYAGE SOUS
FORME DE TABLETTES AYANT DES PROPRIETES DE
DISSOCIATION AMELIOREES**
(54) **LAUNDRY DETERGENT AND CLEANING PRODUCT TABLETS
WITH IMPROVED DISSOCIATION PROPERTIES**

(57) A tablet of compacted particular laundry detergent or cleaning product comprising at least one surfactant, at least one builder and if desired, additional laundry detergent or cleaning product constituents, and further comprising from 2 to 20% by weight of at least one fatty alcohol sulfate, based on the tablet weight and less than 1 % by weight of nonionic surfactants based on the tablet weight and having a melting point below 60°C.

ABSTRACT

A tablet of compacted particular laundry detergent or cleaning product comprising at least one surfactant, at least one builder and if desired,
5 additional laundry detergent or cleaning product constituents, and further comprising from 2 to 20% by weight of at least one fatty alcohol sulfate, based on the tablet weight and less than 1% by weight of nonionic surfactants based on the tablet weight and having a melting point below 60°C.

**LAUNDRY DETERGENT AND CLEANING PRODUCT TABLETS WITH
IMPROVED DISSOCIATION PROPERTIES**

5 The present invention is situated in the field of compact shaped bodies which have wash-active and deter-
sive properties. The invention relates in particular to laundry detergent and cleaning product tablets used for washing textiles in a domestic washing machine, tablets being the common term for shaped bodies of this kind.

10 Laundry detergent and cleaning product tablets are widely described in the prior art and are enjoying increasing receptiveness among users on account of the ease of metering. Tableted laundry detergents and
15 cleaning products have a number of advantages over their powder-form counterparts: they are easier to meter and handle and their compact structure gives advantages in storage and transport. In the patent literature as well, therefore, laundry detergent and
20 cleaning product tablets have been comprehensively described. One problem which occurs again and again in connection with the use of wash-active and deter-
sive tablets is the insufficient disintegration and dissolution rate of the tablets under service
25 conditions. Since sufficiently stable tablets, i.e., dimensionally stable and fracture-resistant tablets, can be produced only by means of relatively high compressive pressures, there is severe compaction of the tablet constituents and, consequently, retarded
30 disintegration of the tablet in the aqueous liquor, and thus excessively slow release of the active substances in the washing or cleaning operation. The retarded disintegration of the tablets has the further disadvantage that customary laundry detergent and
35 cleaning product tablets cannot be rinsed in via the dispenser drawer of domestic washing machines, since the tablets do not break down with sufficient rapidity into secondary particles which are small enough to be

- 2 -

rinsed out of the dispenser drawer into the washing drum.

To overcome the dichotomy between hardness, i.e.,
5 transport and handling stability, and ready disintegration of the tablets, many proposed solutions have been developed in the prior art. One approach, which is known in particular from pharmacy and has expanded into the field of laundry detergent and cleaning product
10 tablets, is the incorporation of certain disintegration aids, which facilitate the ingress of water or swell on ingress of water, and exert a disintegrating action by evolving gas or in some other form. Other proposed solutions from the patent literature describe the compression of premixes of specific particle sizes, the
15 separation of individual ingredients from certain other ingredients, and the coating of individual ingredients or of the entire tablet with binders.

20 For instance, EP-A-0 522 766 (Unilever) discloses tablets of a compacted, particulate laundry detergent composition comprising surfactants, builders and disintegration aids (based on cellulose, for example), at least some of the particles being coated with the
25 disintegrant, which has both a binder effect and a disintegrating effect when the tablets are dissolved in water. This document also points to the general difficulty of producing tablets combining adequate stability with effective solubility. The particle size
30 in the mixture to be compressed is said in this case to be above 200 μm , the intention being that the upper and lower limits of the individual particle sizes differ from one another by not more than 700 μm .

35 Further documents which concern themselves with the production of laundry detergent tablets are EP-A-0 716 144 (Unilever), which describes tablets having an external shell of water-soluble material, and

- 3 -

EP-A-0 711 827 (Unilever), where one ingredient is a citrate having a defined solubility.

5 The use of binders which may develop a disintegrating action (especially polyethylene glycol) is described in EP-A-0 711 828 (Unilever), which describes laundry detergent tablets produced by compressing a particulate laundry detergent composition at temperatures between 28°C and the melting point of the binder material,
10 compression always taking place at below the melting temperature. The examples of that document reveal that the tablets produced in accordance with its teaching have higher fracture strengths if compression is carried out at elevated temperature.

15 Laundry detergent tablets in which individual ingredients are present separately from others are described, inter alia, in EP-A-0 481 793 (Unilever). The laundry detergent tablets disclosed in that document comprise
20 sodium percarbonate, which is spatially separate from all other components that might affect its stability.

In all of the documents of the prior art which deal with laundry detergent and cleaning product tablets the
25 tablets comprise nonionic surfactants in significant amounts. None of said documents deals with improving the solubility of laundry detergent and cleaning product tablets by the deliberate omission of certain ingredients which adversely affect the hardness or
30 disintegration properties of the tablets.

It is an object of the present invention, accordingly, to provide laundry detergent and cleaning product tablets which unite the desired properties of high
35 hardness and mechanical stability with favorable disintegration rates.

It has now been found that laundry detergent tablets of high hardness and yet with an extremely high disintegration rate can be produced if the use of nonionic surfactants having melting points below 60°C is substantially avoided when formulating the laundry detergent and cleaning product recipe.

The invention accordingly provides laundry detergent and cleaning product tablets of compacted particulate laundry detergent and cleaning product, comprising surfactant(s), builder(s), and, if desired, further laundry detergent and cleaning product constituents, wherein the tablets are substantially free from nonionic surfactants having a melting point below 60°C.

Nonionic surfactants which in accordance with the teaching of the present invention or not to be present in the tablets possess melting points of below 60°C and are, for example, alkoxylated alcohols, especially primary alcohols such as fatty alcohols of animal or vegetable origin. In addition to these fatty alcohol alkoxylates, the group of the nonionic surfactants also includes alkyl glycosides of the general formula $RO(G)_x$, where R is a primary straight-chain or methyl-branched aliphatic radical, especially an aliphatic radical methyl-branched in position 2, having 8 to 22, preferably 12 to 18, carbon atoms and G is the symbol for a glucose unit having 5 or 6 carbon atoms, preferably glucose.

Further classes of nonionic surfactants are alkoxylated fatty acid alkyl esters and polyhydroxy fatty acid amides. The use of nonionic surfactants of the amine oxide type, examples being N-cocoalkyl-N,N-dimethylamine oxide and N-tallowalkyl-N,N-dihydroxyethylamine oxide, and of the fatty acid alkanolamides, is also avoided in the context of the present invention.

Although it is possible in accordance with the invention to use nonionic surfactants which possess a melting point above 60°C, it is nevertheless preferred for the tablets as a whole to be substantially free from nonionic surfactants, irrespective of the melting point of the nonionic surfactant.

In the context of the present invention, the term "essentially free from nonionic surfactants" characterizes laundry detergent and cleaning product tablets whose nonionic surfactant content is less than 1% by weight. The reason for this lower limit is that certain ingredients of laundry detergents and cleaning products may be contaminated with nonionic surfactants and yet it may not be desirable to avoid the use of such ingredients. For example, in the preparation of spray-drying base powders containing zeolite, a small amount of nonionic surfactants is added to the slurry that is to be spray-dried. The use of contaminated builders of this kind in which the nonionic surfactant makes no essential contribution to the wash performance of the subsequently produced tablet is therefore possible even in the laundry detergent and cleaning product tablets of the invention. In preferred tablets, the overall nonionic surfactant content, however, is well below the abovementioned upper limit of 1% by weight. Particular preference is given to tablets in which the nonionic surfactant content is below 0.5% by weight, particular preference being given to contents below 0.2% by weight. Most preferred in accordance with the invention, however, are tablets which are totally free from nonionic surfactants, i.e., whose nonionic surfactant content is below the analytical detection limit.

35

In the context of the present invention, the term "nonionic surfactants" expressly does not refer to the pure polyalkylene glycols such as polyethylene glycols

(PEGs) and polypropylene glycols (PPGs). These possess virtually no surface-active properties and are incorporated into laundry detergent and cleaning product tablets as binding assistants and
5 disintegration aids. The PEG and/or PPG content of laundry detergent and cleaning product tablets of the invention may be up to 5% by weight, amounts below 2% by weight being preferred.

10 In order to develop the wash performance, the laundry detergent and cleaning product tablets of the invention comprise surface-active substances from the group of anionic, zwitterionic or cationic surfactants, distinct
15 preference being given to anionic surfactants on economic grounds and on account of their performance spectrum.

Anionic surfactants used are, for example, those of the sulfonate and sulfate type. Suitable surfactants of the
20 sulfonate type are preferably C₉₋₁₃ alkylbenzenesulfonates, olefinsulfonates, i.e., mixtures of alkene- and hydroxyalkanesulfonates, and also disulfonates, as obtained, for example, from C₁₂₋₁₈ monoolefins having a
25 terminal or internal double bond by sulfonation with gaseous sulfur trioxide and subsequently alkaline or acidic hydrolysis of the sulfonation products. Also suitable are alkanesulfonates obtained from C₁₂₋₁₈
alkanes, for example, by sulfochlorination or sulf-
30 oxidation with subsequent hydrolysis or neutralization, respectively. Likewise suitable are the esters of α -sulfo fatty acids (ester sulfonates), e.g., the α -sulfonated methyl esters of hydrogenated coconut, palm kernel or tallow fatty acids.

35 Further suitable anionic surfactants are sulfated fatty acid glycerol esters. By fatty acid glycerol esters are meant the monoesters, diesters and triesters, and also mixtures thereof, as obtained in the preparation by

- esterification of a monoglycerol with from 1 to 3 mol of fatty acid or in the transesterification of triglycerides with from 0.3 to 2 mol of glycerol. Preferred sulfated fatty acid glycerol esters here are the sulfation products of saturated fatty acids having 6 to 22 carbon atoms, examples being those of caproic acid, caprylic acid, capric acid, myristic acid, lauric acid, palmitic acid, stearic acid or behenic acid.
- Preferred alk(en)yl sulfates are the alkali metal salts and, in particular, the sodium salts of the sulfuric monoesters of C₁₂-C₁₈ fatty alcohols, for example, those of coconut fatty alcohol, tallow fatty alcohol, lauryl, myristyl, cetyl or stearyl alcohol, or of the C₁₀-C₂₀ oxo alcohols, and of those monoesters of secondary alcohols of these chain lengths. Preference extends to alkyl(en)yl sulfates of said chain length which contain a synthetic straight-chain alkyl radical prepared on a petrochemical basis and which possess degradation behavior similar to that of the appropriate compounds based on fatty-chemical raw materials. From a detergents standpoint, the C₁₂-C₁₆ alkyl sulfates and C₁₂-C₁₅ alkyl sulfates, and also C₁₄-C₁₅ alkyl sulfates, are preferred. Suitable anionic surfactants also include 2,3-alkyl sulfates, which are prepared, for example, in accordance with US Patents 3,234,258 or 5,075,041 and may be obtained as commercial products of the Shell Oil Company under the name DAN[®].
- The sulfuric monoesters of the straight-chain or branched C₇₋₂₁ alcohols ethoxylated with from 1 to 6 mol of ethylene oxide, such as 2-methyl-branched C₉₋₁₁ alcohols containing on average 3.5 mol of ethylene oxide (EO), or C₁₂₋₁₈ fatty alcohols containing from 1 to 4 EO, are also suitable. Because of their high foaming, they are used in cleaning products only in relatively small amounts: for example, in amounts of from 1 to 5% by weight.

Further suitable anionic surfactants include the salts of alkylsulfosuccinic acid, which are also known as sulfosuccinates or as sulfosuccinic esters and which
5 constitute monoesters and/or diesters of sulfosuccinic acid with alcohols, preferably fatty alcohols and especially ethoxylated fatty alcohols. Preferred sulfosuccinates contain C₈₋₁₈ fatty alcohol radicals or mixtures thereof. Particularly preferred sulfosuccinates include a fatty alcohol radical which is derived
10 from ethoxylated fatty alcohols which per se are non-ionic surfactants (for description, see below). Particular preference is given in turn to sulfosuccinates whose fatty alcohol radicals are derived from ethoxylated fatty alcohols having a narrowed homologue
15 distribution. Likewise, it is also possible to use alk(en)ylsuccinic acid having preferably 8 to 18 carbon atoms in the alk(en)yl chain, or salts thereof.

20 Further anionic surfactants include, in particular, soaps. Saturated fatty acid soaps are suitable, such as the salts of lauric acid, myristic acid, palmitic acid, stearic acid, hydrogenated erucic acid and behenic acid, as in particular are mixtures of soaps derived
25 from natural fatty acids, e.g., coconut, palm kernel or tallow fatty acids.

The anionic surfactants, including the soaps, may be present in the form of their sodium, potassium or ammonium salts and also as soluble salts of organic bases,
30 such as mono-, di- or triethanolamine. Preferably, the anionic surfactants are in the form of their sodium or potassium salts, in particular in the form of the sodium salts.

35

In the context of the present invention, preference is given to laundry detergent and cleaning product tablets containing from 5 to 60% by weight, preferably from 10

to 50% by weight, and in particular from 15 to 30% by weight, of anionic surfactant(s), based in each case on the tablet weight.

5 In connection with the selection of the anionic surfactants used in the laundry detergent and cleaning product tablets of the invention, the freedom of formulation is not restricted by any boundary conditions to be observed. Preferred laundry detergent
10 and cleaning product tablets, however, have a soap content which exceeds 0.2% by weight, based on the overall weight of the tablet. Preferred anionic surfactants for use are the alkylbenzenesulfonates and fatty alcohol sulfates, with preferred laundry deter-
15 gent and cleaning product tablets containing from 2 to 20% by weight, preferably from 5 to 15% by weight, and in particular from 7.5 to 12.5% by weight, of fatty alcohol sulfate(s), based in each case on the tablet weight.

20

In addition to the wash-active substances, builders are the most important ingredients of laundry detergents and cleaning products. In the laundry detergent and cleaning product tablets of the invention all builders
25 commonly used in laundry detergents and cleaning products may be present, i.e., in particular, zeolites, silicates, carbonates, organic cobuilders, and - where there are no ecological prejudices against their use - the phosphates as well.

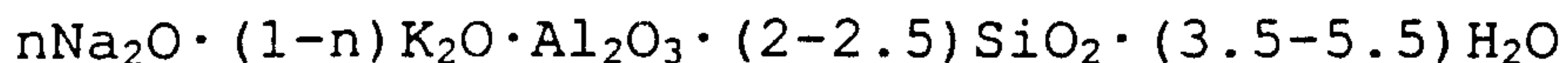
30

Suitable crystalline, layer-forming sodium silicates possess the general formula $\text{NaMSi}_x\text{O}_{2x+1} \cdot \text{H}_2\text{O}$, where M is sodium or hydrogen, x is a number from 1.9 to 4 and y is a number from 0 to 20, and preferred values for x
35 are 2, 3 or 4. Crystalline layered silicates of this kind are described, for example, in European Patent Application **EP-A-0 164 514**. Preferred crystalline layered silicates of the stated formula are those in

which M is sodium and x adopts the values 2 or 3. In particular, both β - and δ -sodium disilicates $\text{Na}_2\text{Si}_2\text{O}_5 \cdot y\text{H}_2\text{O}$ are preferred, β -sodium disilicate being obtainable, for example, by the process described in
5 International Patent Application **WO-A-91/08171**.

It is also possible to use amorphous sodium silicates having an $\text{Na}_2\text{O}:\text{SiO}_2$ modulus of from 1:2 to 1:3.3, preferably from 1:2 to 1:2.8, and in particular from
10 1:2 to 1:2.6, which are dissolution-retarded and have secondary washing properties. The retarded dissolution relative to conventional amorphous sodium silicates may have been brought about by a variety of means: for example, by surface treatment, compounding, compacting,
15 or overdrying. In the context of this invention, the term "amorphous" also embraces "X-ray-amorphous". This means that in X-ray diffraction experiments the silicates do not produce the sharp X-ray reflections typical of crystalline substances but instead at best
20 exhibit one or more maxima of the scattered X-rays, having a width of 2 or more degree units of the diffraction angle. However, particularly good builder properties may very well result if the silicate particles in electron diffraction experiments yield
25 vague or even sharp diffraction maxima. The interpretation of this is that the products contain microcrystalline regions with a size of from 10 to several hundred nm, values of up to max. 50 nm and in particular up to max. 20 nm being preferred. So-called
30 X-ray amorphous silicates of this kind, which likewise possess retarded dissolution relative to the conventional waterglasses, are described, for example, in German Patent Application **DE-A-44 00 024**. Particular preference is given to compacted amorphous silicates,
35 compounded amorphous silicates, and overdried X-ray-amorphous silicates.

The finely crystalline synthetic zeolite used, containing bound water, is preferably zeolite A and/or P. As zeolite P, zeolite MAP[®] (commercial product of Crosfield) is particularly preferred. Also suitable, however, are zeolite X, and also mixtures of A, X and/or P. Also commercially available, and suitable for preferred use in the context of the present invention, is, for example, a cocrystallizate of zeolite X and zeolite A (approximately 80% by weight zeolite X), which is sold by CONDEA Augusta S.p.A. under the brand name VEGOBOND AX[®] and may be described by the formula



The zeolite can be used both as a builder in a granular compound formulation and as a kind of "powdering" for the entire mixture intended for compression, it being usual to utilize both ways of incorporating the zeolite into the premix. Suitable zeolites have an average particle size of less than 10 μm (volume distribution; measurement method: Coulter counter) and contain preferably from 18 to 22% by weight, in particular from 20 to 22% by weight, of bound water.

Of course, the phosphates, which are general knowledge, may also be used as builder substances, provided such use is not to be avoided on ecological grounds. The sodium salts of the orthophosphates, of the pyrophosphates and, in particular, of the tripolyphosphates are especially suitable.

Organic builder substances which may be used are, for example, the polycarboxylic acids that can be used in the form of their sodium salts, such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, sugar acids, amino carboxylic acids, nitrilotriacetic acid (NTA), provided such a use is not objectionable on ecological grounds, and also mixtures

thereof. Preferred salts are the salts of the polycarboxylic acids such as citric acid, adipic acid, succinic acid, glutamic acid, tartaric acid, sugar acids, and mixtures thereof.

5

In order to facilitate the disintegration of highly compacted tablets it is possible to incorporate disintegration aids, known as tablet disintegrants, into them in order to shorten the disintegration times.

10 Tablet disintegrants or disintegration accelerators are, according to Römpf (9th Edition, Vol. 6, p. 4440) and Voigt "*Lehrbuch der pharmazeutischen Technologie*" (6th Edition, 1987, pp. 182-184), auxiliaries which ensure the rapid disintegration of tablets in water or
15 gastric fluid and ensure the release of the drugs in absorbable form.

These disintegrants increase their volume on ingress of water, with on the one hand an increase in the inherent
20 volume (swelling) while on the other hand a pressure may also be produced by way of the release of gases, said pressure causing the tablet to disintegrate into smaller particles. Examples of established disintegration aids are carbonate/citric acid systems, in which
25 context other organic acids may also be used. Swelling disintegration aids are, for example, synthetic polymers such as polyvinylpyrrolidone (PVP) or natural polymers and/or modified natural substances such as cellulose and starch and their derivatives, alginates,
30 or casein derivatives.

Preferred laundry detergent and cleaning product tablets contain from 0.5 to 10% by weight, preferably from 3 to 7% by weight, and in particular from 4 to 6%
35 by weight, of one or more disintegration aids, based in each case on the tablet weight.

Preferred disintegrants used in the context of the present invention are cellulose-based disintegrants, and so preferred laundry detergent and cleaning product tablets contain a cellulose-based disintegrant of this
5 kind in amounts of from 0.5 to 10% by weight, preferably from 3 to 7% by weight, and in particular from 4 to 6% by weight. Pure cellulose has the formal empirical composition $(C_6H_{10}O_5)_n$ and, viewed formally, is a β -1,4-polyacetal of cellobiose, which in turn is
10 composed of two molecules of glucose. Suitable celluloses consist of from about 500 to 5000 glucose units and, accordingly, have average molecular masses of from 50,000 to 500,000. Also suitable for use in the context of the present invention as cellulose-based
15 disintegrants are cellulose derivatives obtainable by polymer-analogous reactions from cellulose. Chemically modified celluloses of this kind include, for example, products from esterifications and etherifications in which hydroxy hydrogen atoms have been substituted.
20 However, celluloses in which the hydroxy groups have been replaced by functional groups which are not attached via an oxygen atom may also be used as cellulose derivatives. The group of the cellulose derivatives embraces, for example, alkali metal
25 celluloses, carboxymethylcellulose (CMC), cellulose esters and cellulose ethers, and aminocelluloses. Said cellulose derivatives are preferably not used as sole cellulose-based disintegrants but instead are used in a mixture with cellulose. The cellulose derivative
30 content of these mixtures is preferably below 50% by weight, with particular preference below 20% by weight, based on the cellulose-based disintegrant. With particular preference, the cellulose-based disintegrant used is pure cellulose free from cellulose derivatives.
35

The cellulose used as a disintegration aid is preferably used not in finely divided form but instead, before being admixed to the premixes intended for

compression, is converted into a coarse form - for example, is granulated or compacted. Laundry detergent and cleaning product tablets comprising disintegrants in granular form or, if appropriate, cogranulated form are described in German Patent Applications **DE 197 09 991** (Stefan Herzog) and **DE 197 10 254** (Henkel) and in International Patent Application **PCT/EP 98/1203** (Henkel). Those documents also contain further details on the preparation of granulated, compacted or cogranulated cellulose disintegrants. The particle sizes of such disintegrants are usually above 200 μm , preferably between 300 and 1600 μm to the extent of at least 90% by weight, and in particular between 400 and 1200 μm to the extent of at least 90% by weight. The relatively coarse cellulose-based disintegration aids mentioned above and described in more detail in the cited documents are preferred for use as disintegration aids in the context of the present invention and are obtainable commercially, for example, under the designation Arbocel[®] TF-30-HG from Rettenmaier.

As a further cellulose-based disintegrant, or as a constituent of that component, it is possible to use microcrystalline cellulose. This microcrystalline cellulose is obtained by partial hydrolysis of celluloses under conditions such that only the amorphous regions (approximately 30% of the total cellulose mass) of the celluloses are attacked and broken down completely, leaving the crystalline regions (approximately 70%) undamaged. Subsequent deaggregation of the microfine celluloses resulting from the hydrolysis yields the microcrystalline celluloses, which have primary particle sizes of approximately 5 μm and can be compacted, for example, to granules having an average particle size of 200 μm .

Laundry detergent and cleaning product tablets further comprising a disintegration aid, preferably a cellulose-based disintegration aid, preferably in granular, cogenerated or compacted form, in amounts of 5 0.5 to 10% by weight, preferably from 3 to 7% by weight, and in particular from 4 to 6% by weight, based in each case on the tablet weight, are particularly preferred in the context of the present invention.

10 Wash-active and deterative tablets are produced by applying pressure to a mixture intended for compression that is located in the cavity of a press. In the simplest case of tablet production, which is called simply tableting below, the mixture to be tableted is 15 compressed directly, i.e., without granulation beforehand. The advantages of this so-called direct tableting are its simple and cost-effective application, since no other process steps and, accordingly, no additional equipment either are required. However, these 20 advantages are also accompanied by disadvantages. For instance, a powder mixture which is to be tableted directly is required to possess sufficient plastic deformability and to have good flow properties; furthermore, it must not exhibit any separation 25 tendencies whatsoever during storage, transport, and the filling of the die. With many mixtures of substances, these three prerequisites can only be managed with extreme difficulty, so that direct tableting, especially for the production of laundry 30 detergent and cleaning product tablets, is seldom employed. The usual route for producing laundry detergent and cleaning product tablets starts, therefore, from pulverulent components ("primary particles"), which by means of suitable techniques are 35 agglomerated or granulated to secondary particles having a greater particle diameter. These granules, or mixtures of different granules, are then mixed with

- 16 -

individual pulverulent adjuvants and passed on for tableting.

In the context of the present invention, preferred
5 laundry detergent and cleaning product tablets are obtained by compressing a particulate premix of one or more sets of surfactant-containing granules and at least one pulverulent component which is admixed subsequently. For the subsequent laundry detergent and
10 cleaning product tablets, it is advantageous if the premix for compression has a bulk density close to that of conventional compact laundry detergents. In particular, it is preferable for the premix for compression to have a bulk density of at least 500 g/l, preferably at least 600 g/l, and in particular more
15 than 700 g/l.

Before the compression of the particulate premix to form laundry detergent and cleaning product tablets,
20 the premix may be "powdered" with finely divided surface treatment agents. This may be of advantage for the constitution and physical properties both of the premix (storage, compression) and of the finished laundry detergent and cleaning product tablets. Finely
25 divided powdering agents are well known in the prior art, with zeolites, silicates or other inorganic salts usually being used. Preferably, however, the premix is "powdered" with finely divided zeolite, preference being given to zeolites of the faujasite type. In the
30 context of the present invention, the term "zeolite of the faujasite type" characterizes all three zeolites which form the faujasite subgroup of the zeolite structure group 4 (compare Donald W. Breck: "Zeolite Molecular Sieves", John Wiley & Sons, New York, London,
35 Sydney, Toronto, 1974, page 92). In addition to zeolite X, therefore, it is also possible to use zeolite Y and faujasite and also mixtures of these compounds, preference being given to pure zeolite X.

Mixtures or cocrystallizates of zeolites of the faujasite type with other zeolites, which need not necessarily belong to the zeolite structure group 4, may also be used as powdering agents, it being advantageous for at least 50% by weight of the powdering agent to consist of a faujasite-type zeolite.

In the context of the present invention, preference is given to laundry detergent and cleaning product tablets consisting of a particulate premix which comprises granular components and pulverulent substances admixed subsequently, where the pulverulent components, or one of the pulverulent components, admixed subsequently is a zeolite of the faujasite type having particle sizes less than 100 μm , preferably less than 10 μm , and in particular less than 5 μm , and accounts for at least 0.2% by weight, preferably at least 0.5% by weight, and in particular more than 1% by weight, of the premix intended for compression.

In addition to the abovementioned constituents, surfactant, builder, and disintegration aid, the laundry detergent and cleaning product tablets of the invention may comprise further ingredients which are customary in laundry detergents and cleaning products, from the group consisting of bleaches, bleach activators, enzymes, fragrances, perfume carriers, fluorescers, dyes, foam inhibitors, silicone oils, antiredeposition agents, optical brighteners, graying inhibitors, color transfer inhibitors, and corrosion inhibitors.

Among the compounds used as bleaches which in water produce H_2O_2 , particular significance is possessed by sodium perborate tetrahydrate and sodium perborate monohydrate. Examples of other bleaches which can be used are sodium percarbonate, peroxyphosphates, citrate perhydrates, and H_2O_2 -donating peracidic salts

or peracids, such as perbenzoates, peroxophthalates, diperazelaic acid, phthaloiminoperacid or diperdodecanedioic acid.

5 In order to achieve an improved bleaching action when washing at temperatures of 60°C and below, it is possible to incorporate bleach activators as the sole constituent or as an ingredient of component (b). Bleach activators used may be compounds which under
10 perhydrolysis conditions give rise to aliphatic peroxocarboxylic acids having preferably 1 to 10 carbon atoms, especially 2 to 4 carbon atoms, and/or substituted or unsubstituted perbenzoic acids. Suitable substances are those which carry O-acyl and/or N-acyl
15 groups of the stated number of carbon atoms, and/or substituted or unsubstituted benzoyl groups. Preference is given to polyacylated alkylenediamines, especially tetraacetylenediamine (TAED), acylated triazine derivatives, especially 1,5-diacetyl-2,5-dioxohexa-
20 hydro-1,3,5-triazine (DADHT), acylated glycolurils, especially tetraacetylglycoluril (TAGU), N-acyl imides, especially N-nonanoylsuccinimide (NOSI), acylated phenolsulfonates, especially n-nonanoyl- or isononanoyloxybenzenesulfonate (n- or iso-NOBS), carboxylic
25 anhydrides, especially phthalic anhydride, acylated polyhydric alcohols, especially triacetin, ethylene glycol diacetate, and 2,5-diacetoxy-2,5-dihydrofuran.

In addition to the conventional bleach activators, or
30 in their place, it is also possible to incorporate what are known as bleaching catalysts into the tablets. These substances comprise bleach-boosting transition metal salts or transition metal complexes such as, for example, Mn-, Fe-, Co-, Ru- or Mo-salen complexes or
35 -carbonyl complexes. In addition, Mn, Fe, Co, Ru, Mo, Ti, V and Cu complexes with N-containing tripod ligands, and also Co-, Fe-, Cu- and Ru-amine complexes, may be used as bleaching catalysts.

Suitable enzymes are those from the class of the proteases, lipases, amylases, cellulases, and/or mixtures thereof. Especially suitable enzymatic active substances are those obtained from bacterial strains or fungi, such as *Bacillus subtilis*, *Bacillus licheniformis* and *Streptomyces griseus*. Preference is given to the use of proteases of the subtilisin type, and especially to proteases obtained from *Bacillus lentus*. Of particular interest in this context are mixtures of enzymes; for example, of protease and amylase or protease and lipase or protease and cellulase, or of cellulase and lipase, or of protease, amylase and lipase or protease, lipase and cellulase, but especially mixtures containing cellulase. Peroxidases or oxidases have also proven suitable in some cases. The enzymes may be adsorbed on carrier substances and/or embedded in coat substances in order to protect them against premature decomposition. The proportion of the enzymes, enzyme mixtures or enzyme granules in the tablets of the invention may amount, for example, to from about 0.1 to 5% by weight, preferably from 0.1 to about 2% by weight.

In addition, the laundry detergent and cleaning product tablets may also include components which have a positive effect on the capacity for oil and fat to be washed off from textiles (these components being known as soil repellents). This effect is particularly marked if a textile which has already been washed several times beforehand with a laundry detergent of the invention comprising this oil- and fat-dissolving component becomes soiled. The preferred oil- and fat-dissolving components include, for example, nonionic cellulose ethers such as methylcellulose and methylhydroxypropylcellulose having a methoxyl group fraction of from 15 to 30% by weight and a hydroxypropoxyl group fraction of from 1 to 15% by

weight, based in each case on the nonionic cellulose ether, and also the prior art polymers of phthalic acid and/or of terephthalic acid and/or of derivatives thereof, especially polymers of ethylene terephthalates and/or polyethylene glycol terephthalates, or anionically and/or nonionically modified derivatives of these. Of these, particular preference is given to the sulfonated derivatives of phthalic acid polymers and of terephthalic acid polymers.

10

As optical brighteners, the tablets may comprise derivatives of diaminostilbenedisulfonic acid and/or its alkali metal salts. Suitable brighteners are, for example, salts of 4,4'-bis(2-anilino-4-morpholino-1,3,5-triazinyl-6-amino)stilbene-2,2'-disulfonic acid or compounds of similar construction which instead of the morpholino group carry a diethanolamino group, a methylamino group, an anilino group, or a 2-methoxyethylamino group. In addition, brighteners of the substituted diphenylstyryl type may be present, examples being the alkali metal salts of 4,4'-bis(2-sulfostyryl)biphenyl, of 4,4'-bis(4-chloro-3-sulfostyryl)biphenyl, or of 4-(4-chlorostyryl)-4'-(2-sulfostyryl)biphenyl. Mixtures of the abovementioned brighteners may also be used.

Dyes and fragrances are added to the compositions of the invention in order to enhance the aesthetic appeal of the products and to provide the user not only with the wash performance but also with a visually and sensorially "typical and unmistakable" product. As perfume oils and fragrances it is possible to use individual odorant compounds, e.g., the synthetic products of the type of the esters, ethers, aldehydes, ketones, alcohols, and hydrocarbons. Odorant compounds of the ester type are, for example, benzyl acetate, phenoxyethyl isobutyrate, p-tert-butylcyclohexyl acetate, linalyl acetate, dimethylbenzylcarbinyl

acetate, phenylethyl acetate, linalyl benzoate, benzyl formate, ethyl methylphenylglycinate, allyl cyclohexylpropionate, styrallyl propionate, and benzyl salicylate. The ethers include, for example, benzyl ethyl ether; the aldehydes include, for example, the linear alkanals having 8 to 18 carbon atoms, citral, citronellal, citronellyloxyacetaldehyde, cyclamen aldehyde, hydroxycitronellal, lilial and bourgeonal; the ketones include, for example, the ionones, α -isomethylionone and methyl cedryl ketone; the alcohols include anethole, citronellol, eugenol, geraniol, linalool, phenylethyl alcohol and terpineol; and the hydrocarbons include primarily the terpenes such as limonene and pinene. Preference is given, however, to the use of mixtures of different odorants which together produce an appealing fragrance. Such perfume oils may also comprise natural mixtures of odorants, as are obtainable from plant sources, e.g. pine oil, citrus oil, jasmine oil, patchouli oil, rose oil, or ylang-ylang oil. Likewise suitable are clary sage oil, camomile oil, clove oil, balm oil, mint oil, cinnamon leaf oil, lime blossom oil, juniperberry oil, vetiver oil, olive oil, galbanum oil and labdanum oil, and also orange blossom oil, neroliol, orange peel oil, and sandalwood oil.

The dye content of the tablets of the invention is usually less than 0.01% by weight, while fragrances may account for up to 2% by weight of the overall formulation.

The fragrances may be incorporated directly into the compositions of the invention; alternatively, it may be advantageous to apply the fragrances to carriers which reinforce the adhesion of the perfume on the laundry and ensure persistent fragrance of the textiles by means of a slower release of fragrance. Examples of such carrier materials which have proven suitable are

cyclodextrins, in which case, in addition, the cyclodextrin-perfume complexes may also be coated with further auxiliaries.

5 In order to enhance the aesthetic appeal of the compositions of the invention, they may be colored with suitable dyes. Preferred dyes, whose selection causes no difficulty whatsoever to the skilled worker, possess a high level of storage stability and insensitivity to
10 the other ingredients of the compositions, and to light, and do not possess any pronounced substantivity with respect to textile fibers, so that they do not stain said fibers.

15 The tablets of the invention are produced first of all by the dry mixing of the constituents, some or all of which may have been pregranulated, and the subsequent shaping - in particular, compressing to form tablets - which can be done by recourse to conventional
20 processes. To produce the tablets of the invention, the premix is compacted in a so-called die between two punches to form a solid compact. This process, referred to below for short as tableting, is divided into four sections: metering, compaction (elastic deformation),
25 plastic deformation, and ejection.

First of all, the premix is introduced into the die, the amount and thus the weight and form of the resulting tablet being determined by the position of the
30 lower punch and the shape of the compression tool. Consistent metering even at high tablet throughputs is achieved preferably by volumetric metering of the premix. In the subsequent course of tableting, the upper punch contacts the premix and is lowered further
35 in the direction of the lower punch. Under this compaction, the particles of the premix are pressed closer to one another, with a continual reduction in the cavity volume within the filling between the

punches. When a certain position of the upper punch is reached (and thus when a certain pressure on the premix is reached), plastic deformation begins, in which the particles coalesce and the tablet is formed. Depending on the physical properties of the premix, some of the premix particles are also crushed, and at even higher pressures, sintering of the premix occurs. As the compression speed increases, i.e., with high throughputs, the phase of elastic deformation becomes shorter and shorter, so that the resulting tablets may have larger or smaller cavities. In the final step of tableting, the finished tablet is ejected from the die by the lower punch and is conveyed away by subsequent transport devices. At this point in time, only the weight of the tablet is ultimately fixed, since owing to physical processes (re-expansion, crystallographic effects, cooling, etc.) the compacts may still change their shape and size.

Tableting takes place in commercially customary tableting presses, which may in principle be equipped with single or double punches. In the latter case the upper punch is not used alone to build up pressure; the lower punch, as well, moves toward the upper punch during the compression operation, while the upper punch presses downward. For small production volumes it is preferred to use eccentric tableting presses, where the punch or punches is or are fastened to an eccentric disk which is itself mounted on an axle with a particular speed of revolution. The movement of these compressing punches is comparable with the way in which a customary four-stroke engine operates. Compression may take place with one upper punch and one lower punch, or else a plurality of punches may be fastened to one eccentric disk, in which case the number of die bores is increased accordingly. The throughputs of eccentric presses vary, depending on model, from several hundred to a maximum of 3000 tablets per hour.

For larger throughputs, rotary tableting presses are chosen, in which a larger number of dies is arranged in a circle on a so-called die table. Depending on model, the number of dies varies between 6 and 55, with larger dies also being obtainable commercially. Each die on the die table is allocated an upper and lower punch, it being possible in turn for the compressive pressure to be built up actively only by the upper punch or lower punch or else by both punches. The die table and the punches move around a common, vertical axis, the punches being brought into the filling, compaction, plastic deformation, and ejection positions, during revolution, with the aid of raillike cam tracks. At the positions necessitating a considerable lifting or lowering of the punches (filling, compaction, ejection), these cam tracks are assisted by additional low-pressure pieces, low tension rails, and discharge tracks. The die is filled by way of a rigid feed device, known as the filling shoe, which is connected to a reservoir vessel for the premix. The compressive pressure on the premix can be adjusted individually for upper punch and lower punch by way of the compression paths, the buildup of pressure taking place by the rolling of the punch shaft heads past adjustable pressure rolls.

In order to increase the throughput, rotary presses may also be provided with two filling shoes, in which case only a half-circle need be traversed in order to produce one tablet. For the production of two-layer and multilayer tablets, a plurality of filling shoes are arranged in series, without the slightly compressed first layer being ejected before the subsequent filling. By means of an appropriate process regime, it is also possible in this way to produce laminated tablets and inlay tablets having a structure like that of an onion skin, where in the case of the inlay

tablets the top face of the core, or of the core layers, is not covered and therefore remains visible. Rotary tableting presses may also be equipped with single or multiple tools, so that, for example, an
5 outer circle with 50 bores and an inner circle with 35 bores are used simultaneously for compression. The throughputs of modern rotary tableting presses amount to more than one million tablets per hour.

10 Tableting machines suitable in the context of the present invention are obtainable, for example, from the following companies: Apparatebau Holzwarth GbR, Asperg, Wilhelm Fette GmbH, Schwarzenbek, Hofer GmbH, Weil, KILIAN, Cologne, KOMAGE, Kell am See, KORSCH Pressen
15 GmbH, Berlin, Mapag Maschinenbau AG, Berne (CH) and Courtoy N.V., Halle (BE/LU). Particularly suitable, for example, is the hydraulic double-pressure press HPF 630 from LAEIS, D.

20 The tablets may be manufactured in predetermined three-dimensional forms and predetermined sizes. Suitable three-dimensional forms include virtually all practicable designs; i.e., for example, bar forms, rod or ingot forms, cubes, blocks, and corresponding three-
25 dimensional elements having planar side faces, and also, in particular, cylindrical designs with a circular or oval cross section. This last embodiment embraces the presentation form ranging from the tablet through to compact cylinders having a height-to-
30 diameter ratio of more than 1.

The portioned compacts may in each case be designed as individual elements, separated from one another, corresponding to the predetermined dosing amount of the
35 laundry detergents and/or cleaning products. Likewise, however, it is possible to design compacts which combine a plurality of such mass units in one compact, with, in particular, predefined intended breakage

points providing for easy separation of smaller, portioned units. For the use of textile detergents in machines of the type customary in Europe, with a horizontally disposed mechanism, it may be judicious to design the portioned compacts as tablets, in cylinder or block form, preference being given to a diameter/-height ratio in the range from about 0.5:2 to 2:0.5. Commercially customary hydraulic presses, eccentric presses or rotary presses are suitable apparatus in particular for producing compacts of this kind.

The three-dimensional form of another embodiment of the tablets is adapted in its dimensions to the dispenser drawer of commercially customary domestic washing machines, so that the tablets can be metered directly, without a metering aid, into the dispenser drawer, where they dissolve during the rinsing-in operation. It is, however, also possible of course to use the laundry detergent tablets with a metering aid, and this is preferred in the context of the present invention.

Another preferred tablet which may be produced has a sheetlike or barlike structure with, in alternation, long, thick and short, thin segments, so that individual segments can be broken off from this "slab" at the intended breakage points, represented by the short, thin segments, and introduced into the machine. This principle of the laundry detergent "bar" may also be realized in other geometric forms, an example being vertical triangles connected to one another along only one of their sides.

Yet another possibility, however, is not to compress the various components into a uniform tablet but instead to produce tablets having a plurality of layers, i.e., at least two layers. In this case it is also possible for these different layers to have different dissolution rates. This may result in advan-

tageous performance properties of the tablets. If, for example, the tablets include components which have adverse effects on one another, then it is possible to integrate one component into the faster-dissolving layer and the other component into a slower-dissolving layer, so that the first component has already reacted when the second goes into solution. The layer structure of the tablets may be in the form of a stack, with the inside layer(s) at the edges of the tablet dissolving even when the outside layers have not yet dissolved completely; alternatively, the inside layer(s) may be enveloped completely by the respective layer(s) lying further out, which prevents the premature dissolution of constituents of the inside layer(s).

15 In a further-preferred embodiment of the invention, a tablet consists of at least three layers, i.e., two outside layers and at least one inside layer, and a peroxy bleach is present at least in one of the inside layers, while in the stacklike tablet the two outer layers and in the shell-like tablet the outermost layers are free from peroxy bleach. It is also possible, furthermore, to separate peroxy bleach and any bleach activators and/or enzymes present from one another, spatially, in one tablet. Multilayer tablets of this kind have the advantage that they can be used not just by way of a dispenser drawer or by way of a dosing apparatus which is placed in the wash liquor; rather, it is also possible in such cases to place the tablet into the machine in direct contact with the textiles, without running the risk of spotting by bleaches and the like.

Similar effects can also be achieved by coating individual constituents of the laundry detergent and cleaning product composition intended for compression, or of the entire tablet. For this purpose, the tablets to be coated may be sprayed, for example, with aqueous

solutions or emulsions, or may receive a coating by the technique of melt coating.

Following compression, the laundry detergent and cleaning product tablets possess high stability. The fracture strength of cylindrical tablets can be ascertained by way of the parameter of the diametral fracture stress. This can be determined in accordance with

10

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

In this formula, σ is the diametral fracture stress (DFS) in Pa, P is the force in N which leads to the pressure exerted on the tablet, which pressure causes the fracture of the tablet, D is the tablet diameter, in meters, and t is the tablet height.

15

Examples:

20

By blending surfactant-containing granules with pulverulent preparation components, premixes were prepared which were compressed to laundry detergent tablets in a Korsch tableting press. The compressive pressure was adjusted to give in each case two series of tablets differing in their hardness.

25

The surfactant granules which lead to the tablets E1 and E2 of the invention contain no nonionic surfactant, while comparison granules, which on compression give the tablets V1 and V2, contains 5.7% by weight of nonionic surfactant (= 3.5% by weight, based on the tablet). The tablets E1 and E2 and, respectively, V1 and V2 differ only in their hardness, not in their composition. The composition of the surfactant granules, and the composition of the premixes intended

35

for compression (and thus of the tablets), are shown in Tables 1 and 2.

Table 1: Composition of the surfactant granules [% by weight]

	Granules for E1/2	Granules for V1/2
C ₉₋₁₃ alkylbenzenesulfonate	21.2	18.6
C ₁₂₋₁₈ fatty alcohol containing 7 EO	-	5.7
C ₁₂₋₁₈ fatty alcohol sulfate	8.5	5.4
Soap	1.6	1.6
Optical brightener	0.3	0.3
Sodium carbonate	17.0	16.6
Sodium silicate	5.6	5.4
Acrylic acid-maleic acid copolymer	5.6	5.4
Zeolite A (anhydrous active substance)	28.5	29.9
Na 1,1-hydroxyethanediphosphonate	0.8	0.8
Water, salts	Remainder	Remainder

Table 2: Composition of the premix [% by weight]:

Surfactant granules	61.3	61.3
Zeolite	2.0	2.0
Sodium perborate monohydrate	23.7	23.7
TAED	2.4	2.4
Foam inhibitor	4.7	4.7
Polyacrylate	1.4	1.4
Perfume	0.5	0.5
Disintegration aid (cellulose)*	4.0	4.0

10

* Compacted cellulose (particle size: 90% by weight > 400 µm)

The hardness of the tablets was measured by deformation of the tablet until it fractures, with the force acting

15

- 30 -

on the side faces of the tablet and the maximum force withstood by the tablet being measured.

5 To determine the tablet disintegration, the tablet was placed in a glass beaker with water (600 ml of water, temperature 30°C), and the time taken for the tablet to undergo complete disintegration was measured.

The experimental data are shown in Table 3:

10

Table 3: Laundry detergent tablets [physical data]

Tablet	E1	V1	E2	V2
Tablet hardness	57 N	53 N	78 N	68 N
Tablet decomposition	20 sec	120 sec	45 sec	> 120 sec

- 31 -

CLAIMS

1. A tablet of compacted particular laundry detergent or cleaning product comprising at least one surfactant, at least one builder and if desired, additional laundry detergent or cleaning product constituents, and further comprising from 2 to 20% by weight of at least one fatty alcohol sulfate, based on the tablet weight and less than 1% by weight of nonionic surfactants based on the tablet weight and having a melting point below 60°C.
2. The tablet as claimed in claim 1, which is essentially free from nonionic surfactants.
3. The tablet as claimed in either of claims 1 or 2, wherein from 15 to 30% by weight of at least one anionic surfactant based in each case on the tablet weight is present.
4. The tablet as claimed in any of claims 1 to 3, wherein from 5 to 15% by weight, of at least one fatty alcohol sulfate based in each case on the tablet weight is present.
5. The tablet as claimed in any of claims 1 to 3, wherein at least one fatty alcohol sulfate is present in an amount of from 7.5 to 12.5% by weight.
6. The tablet as claimed in any of claims 1 to 5, wherein comprises from 0.5 to 10% by weight, of a disintegration aid based on the tablet weight is present.
7. The tablet as claimed in any of claim 6, wherein the amount is from 1 to 6% by weight based on the tablet weight.
8. The tablet as claimed in claim 6, wherein the amount is from 3 to 5% by weight based on the tablet weight.
9. The tablet as claimed in any one of claims 6 to 8, which comprises as disintegration aid a cellulose-based disintegration aid.

- 32 -

10. The tablet as claimed in any one of claims 1 to 9, obtained by compressing a particulate premix comprising at least one set of surfactant-containing granules and at least one subsequently admixed pulverulent component.
- 5
11. The tablet as claimed in claim 10, wherein the premix for compression has a bulk density of at least 500 g/l.
12. The tablet as claimed in claim 11, wherein the bulk density is at least 600 g/l.
- 10
13. The tablet as claimed in claim 11, wherein the bulk density is more than 700g/l.
14. The tablet as claimed in any one of claims 10 to 13, wherein the subsequently admixed, or one of the subsequently admixed, pulverulent components is a zeolite of the faujasite type having particle sizes less than 100 μm and accounts for at least 0.2% by weight, of the premix intended for compression.
- 15
15. The tablet as claimed in claim 14, wherein the faujasite type has particle sizes of less than 10 μm .
- 20
16. The tablet as claimed in claim 14, wherein the faujasite type has particle sizes of less than 5 μm .
- 25
17. The tablet as claimed in claims 14 to 16, wherein the amount is at least 0.5% by weight.
18. The tablet as claimed in claim 14 to 17, wherein the amount is more than 1% by weight.
- 30
19. The tablet as claimed in any one of claims 1 to 18, further comprising one or more substances from the group consisting of builders, bleaches, bleach activators, enzymes, pH standardizers, fragrances, perfume carriers, fluorescers, dyes, foam inhibitors, silicone oils, antiredeposition agents, optical brighteners, graying inhibitors, color transfer inhibitors, and corrosion inhibitors.
- 35