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(54) **TABLETTES DE DETERGENT POLYPHASIQUES**

(54) **MULTIPHASE DETERGENT TABLETS**

(57) The invention relates to two-phase or multiphase detergent tablets of compacted particulate detergent comprising surfactant(s), builder(s) and optionally other detergent ingredients, in which the surfactant content of the individual phases of the tablets varies by no more than 1.5% by weight, based on the weight of the individual phase. By virtue of this very substantial adaptation of the surfactant contents of the individual phases of the tablet, multiphase tablets with an excellent property profile are obtained.

Abstract

The invention relates to two-phase or multiphase detergent tablets of compacted particulate detergent comprising surfactant(s), builder(s) and optionally other detergent ingredients, in which the surfactant content of the individual phases of the tablets varies by no more than 1.5% by weight, based on the weight of the individual phase. By virtue of this very substantial adaptation of the surfactant contents of the individual phases of the tablet, multiphase tablets with an excellent property profile are obtained.

MULTIPHASE DETERGENT TABLETS

Field of the Invention

This invention relates generally to multiphase detergent tablets. More particularly, the invention relates to multiphase detergent tablets which are used for washing laundry in a domestic washing machine and which are referred to in short as detergent tablets.

Background of the Invention

By virtue of the ease with which they can be dosed and other advantages in regard to packaging, transportation and storage, tablets afford a number of advantages which make it appear desirable to produce detergents also in tablet form. A broad prior art exists on the subject of detergent tablets, being concerned in particular with overcoming a major problem of tablets, namely the dichotomy between the hardness of tablets on the one hand and their disintegration rate on the other hand. Adequate hardness is essential for the packaging, storage, transportation and handling of tablets while their disintegration properties critically influence the washing process and sufficiently rapid disintegration is absolutely essential for the formation of a suitably concentrated wash liquor.

The problem of finding a technically reasonable compromise between hardness and disintegration is further complicated in the case of multiphase tablets. It can be of advantage with the washing process in mind to separate certain detergent ingredients from one another. However, such separation does lead to differences in the physical property profiles of the various phases in the tablet. Thus, in the extreme case, inter-phase adhesion can diminish to such an extent that multiphase tablets can no longer be produced. The effect of an excessive difference in hardness between different phases would be that certain phases would be damaged to a greater extent during packaging, transportation and handling than other phases. In addition, excessive differences between the disintegration

and dissolving rates of individual phases would also be undesirable because otherwise active ingredients from the more slowly disintegrating or dissolving phase would not be available to the washing process.

Accordingly, it is crucially important in the case of multiphase detergent tablets for all the phases to adhere to one another and to show adequate and comparable hardness and a sufficiently rapid and identical disintegration and dissolving profile. No proposed solutions to these problems are described in the prior art.

Detergent tablets in which individual ingredients are separated from others are also described in **EP-A-0 481 793** (Unilever). The detergent tablets disclosed in this document contain sodium percarbonate which is separated from all other components which could affect its stability. The document in question does not mention hardness and/or disintegration as a function of phase composition.

EP-A-0 466 485 (Unilever) describes detergent tablets produced by tabletting two types of surfactant-containing granules. One type contains the total quantity of anionic surfactants while the second type is preferably free from anionic surfactants. This document also does not mention hardness and/or disintegration as a function of phase composition.

Summary of the Invention

Now, the problem addressed by the present invention was to provide multiphase detergent tablets which would overcome the disadvantages mentioned above. More particularly, the invention sought to provide multiphase detergent tablets which would have high hardness values and high disintegration and dissolving rates in all phases.

It has now been found that multiphase detergent tablets with an excellent property profile can be produced providing the surfactant contents in the individual phases are very largely adapted to one another.

Accordingly, the present invention relates to two or more phase

detergent tablets of compacted particulate detergent comprising surfactant(s), builder(s) and optionally other detergent ingredients, characterized in that the surfactant content of the individual phases of the tablets varies by no more than 1.5% by weight, based on the weight of the individual phase.

Detailed Description of the Invention

In the context of the present invention, the variation of the surfactant content by no more than 1.5% by weight, based on the weight of the individual phases, means that the absolute values of the surfactant content in the phases vary by no more than 1.5% by weight. If, therefore, one phase contains 20% by weight of surfactant(s), the surfactant content of the other phase(s) must be selected so that the range of variation about the value 20 is at most 1.5% by weight. In other words, the percentage figure for the surfactant content of the phase with the lower surfactant content is subtracted from that of the phase with the higher surfactant content, the result from phase to phase having to be ≤ 1.5.

In preferred detergent tablets, the surfactant content of the individual phases varies by less than 1.5% by weight. Preferred detergent tablets are characterized in that the surfactant content of the individual phases of the tablet differs by no more than 1% by weight, based on the weight of the individual phase.

Detergent tablets in which the surfactant content of the individual phases of the tablet is identical are particularly preferred. Since the products mentioned are industrial products which are produced in batches of several tonnes for individual weights of normally below 100 grams, a slight variation in the surfactant content of individual phases cannot be completely ruled out. Accordingly, in the context of the invention, an "identical" surfactant content in the individual phases exists even when variations of a few tenths % by weight are present.

According to the invention, the individual phases of the tablets may assume various three-dimensional forms. The most simple embodiment is a two-layer or multilayer tablet, each layer of the tablet representing one phase. However, it is also possible in accordance with the invention to produce multiphase tablets in which individual phases assume the form of inclusions in (an)other phase(s). Besides so-called "ring/core" tablets, jacket tablets or combinations of the embodiments mentioned are also possible. Examples of multiphase tablets can be found in the drawings of **EP-A-0 055 100** (Jeyes) which describes toilet cleaning blocks. Technically the most common form of multiphase tablets are two-layer or multilayer tablets. According to the invention, therefore, the phases of the tablet are preferably in the form of layers.

According to the invention, it is crucial that the surfactant content of the individual phases of the tablet vary by no more than 3% by weight, based on the weight of the individual phase. Determination of the surfactant content is based on the sum of the surfactants present in the particular phase, irrespective of the type of surfactant involved. If one phase contains anionic and nonionic surfactants, for example, the total surfactant content of the phase is the sum of the quantities of anionic and nonionic surfactants.

The surfactants may be incorporated in the individual phases of the tablet in pure form. This is readily possible, for example, in the case of soaps or other readily processable surfactants. With many surfactants, however, it is advisable to incorporate surfactant compounds rather than the pure surfactants. These compounds – which should have high surfactant contents according to the particular application – may be produced by conventional processes, such as spray drying, granulation or compounding. A combination of several batches of surfactant granules or a combination of surfactant granules with pure surfactants is of course also

possible.

According to the invention, the surfactant(s) are preferably introduced into the phases of the tablets through surfactant-containing granules.

In other embodiments of the present invention, different surfactant granules may be used for each phase. However, each phase may also derive its surfactant content from the same granules which are therefore present in all phases of the tablet. Another preferred embodiment of the invention is characterized in that the same surfactant granules are used in all phases of the tablets.

Now, the most simple possible embodiment of the present invention is a two-phase tablet in which the phases are present as layers and in which the same surfactant granules are used in the two layers. These tablets of two layers containing the same surfactant granules can readily be produced in conventional tablet presses.

Anionic, nonionic, cationic and/or amphoteric surfactants or mixtures thereof may be used in the detergent tablets according to the invention. Mixtures of anionic and nonionic surfactants are preferred from the applicational point of view. The tablets have a total surfactant content of 5 to 60% by weight, based on tablet weight, surfactant contents of more than 15% by weight being preferred.

Suitable anionic surfactants are, for example, those of the sulfonate and sulfate type. Suitable surfactants of the sulfonate type are preferably C_{9-13} alkyl benzenesulfonates, olefin sulfonates, i.e. mixtures of alkene and hydroxyalkane sulfonates, and the disulfonates obtained, for example, from C_{12-18} monoolefins with an internal or terminal double bond by sulfonation with gaseous sulfur trioxide and subsequent alkaline or acidic hydrolysis of the sulfonation products. Other suitable surfactants of the sulfonate type are the alkane sulfonates obtained from C_{12-18} alkanes, for example by

sulfochlorination or sulfoxidation and subsequent hydrolysis or neutralization. The esters of α-sulfofatty acids (ester sulfonates), for example the α-sulfonated methyl esters of hydrogenated coconut oil, palm kernel oil or tallow fatty acids, are also suitable.

Other suitable anionic surfactants are sulfonated fatty acid glycerol esters. Fatty acid glycerol esters in the context of the present invention are the monoesters, diesters and triesters and mixtures thereof which are obtained where production is carried out by esterification of a monoglycerol with 1 to 3 moles of fatty acid or in the transesterification of triglycerides with 0.3 to 2 moles of glycerol. Preferred sulfonated fatty acid glycerol esters are the sulfonation products of saturated fatty acids containing 6 to 22 carbon atoms, for example 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 acid semiesters of C_{12-18} fatty alcohols, for example cocofatty alcohol, tallow fatty alcohol, lauryl, myristyl, cetyl or stearyl alcohol, or C_{10-20} oxoalcohols and the corresponding semiesters of secondary alcohols with the same chain length. Other preferred alk(en)yl sulfates are those with the chain length mentioned which contain a synthetic, linear alkyl chain based on a petrochemical and which are similar in their degradation behavior to the corresponding compounds based on oleochemical raw materials. C_{12-16} alkyl sulfates, C_{12-15} alkyl sulfates and C_{14-15} alkyl sulfates are preferred from the point of view of washing technology. Other suitable anionic surfactants are 2,3-alkyl sulfates which may be produced, for example, in accordance with **US 3,234,258** or **US 5,075,041** and which are commercially obtainable as products of the Shell Oil Company under the name of DAN®.

The sulfuric acid monoesters of linear or branched C_{7-21} alcohols ethoxylated with 1 to 6 moles of ethylene oxide, such as 2-methyl-branched

C_{9-11} alcohols containing on average 3.5 moles of ethylene oxide (EO) or C_{12-18} fatty alcohols containing 1 to 4 EO, are also suitable. In view of their high foaming capacity, they are only used in relatively small quantities, for example in quantities of 1 to 5% by weight, in dishwashing detergents.

Other suitable anionic surfactants are the salts of alkyl sulfosuccinic acid which are also known as sulfosuccinates or as sulfosuccinic acid esters and which represent monoesters and/or diesters of sulfosuccinic acid with alcohols, preferably fatty alcohols and, more particularly, ethoxylated fatty alcohols. Preferred sulfosuccinates contain C_{8-18} fatty alcohol residues or mixtures thereof. Particularly preferred sulfosuccinates contain a fatty alcohol residue derived from ethoxylated fatty alcohols which, considered in isolation, represent nonionic surfactants (for a description, see below). Of these sulfosuccinates, those of which the fatty alcohol residues are derived from narrow-range ethoxylated fatty alcohols are particularly preferred. Alk(en)yl succinic acid preferably containing 8 to 18 carbon atoms in the alk(en)yl chain or salts thereof may also be used.

Other suitable anionic surfactants are, in particular, soaps. Suitable soaps are saturated fatty acid soaps, such as the salts of lauric acid, myristic acid, palmitic acid, stearic acid, hydrogenated erucic acid and behenic acid, and soap mixtures derived in particular from natural fatty acids, for example coconut oil, palm kernel oil or tallow fatty acids.

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

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 of ethylene oxide (EO) per

rnole of alcohol, in which the alcohol component may be linear or, preferably, methyl-branched in the 2-position or may contain linear and rnethyl-branched residues in the form of the mixtures typically present in oxoalcohol residues. However, alcohol ethoxylates containing linear residues of alcohols of native origin with 12 to 18 carbon atoms, for example coconut oil, palm oil, tallow fatty or oleyl alcohol, and on average 2 to 8 EO per mole of alcohol are particularly preferred. Preferred ethoxylated alcohols include, for example, C_{12-14} alcohols containing 3 EO or 4 EO, C_{9-11} alcohol containing 7 EO, C_{13-15} alcohols containing 3 EO, 5 EO, 7 EO or 8 EO, C_{12-18} alcohols containing 3 EO, 5 EO or 7 EO and mixtures thereof, such as mixtures of C_{12-14} alcohol containing 3 EO and C_{12-18} alcohol containing 5 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). In addition to these nonionic surfactants, fatty alcohols containing more than 12 EO may also be used, examples including tallow fatty alcohol containing 14 EO, 25 EO, 30 EO or 40 EO.

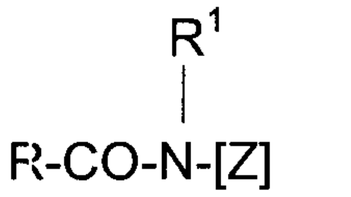
Another class of nonionic surfactants which may advantageously be used are alkyl glycosides corresponding to the general formula $RO(G)_x$ where R is a primary linear or methyl-branched, more particularly 2-methyl-branched, aliphatic radical containing 8 to 22 and preferably 12 to 18 carbon atoms and G stands for a glycose unit containing 5 or 6 carbon atoms, preferably glucose. The degree of oligomerization x, which indicates the distribution of monoglycosides and oligoglycosides, is a number of 1 to 10, preferred values for x being 1.2 to 1.4.

Another class of preferred nonionic surfactants which may be used either as sole nonionic surfactant or in combination with other nonionic surfactants are alkoxylated, preferably ethoxylated or ethoxylated and

propoxylated, fatty acid alkyl esters preferably containing 1 to 4 carbon atoms in the alkyl chain, more especially the fatty acid methyl esters which are described, for example, in Japanese patent application **JP 58/217598** or which are preferably produced by the process described in International patent application **WO-A-90/13533**.

Nonionic surfactants of the amine oxide type, for example N-cocoalkyl-N,N-dimethylamine oxide and N-tallowalkyl-N,N-dihydroxyethyl-amine oxide, and the fatty acid alkanolamide type are also suitable. The quantity in which these nonionic surfactants are used is preferably no more than the quantity in which the ethoxylated fatty alcohols are used and, more preferably, no more than half that quantity.

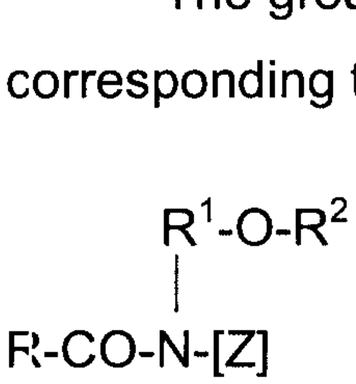
Other suitable surfactants are polyhydroxyfatty acid amides corresponding to formula (I):



(I)

in which RCO is an aliphatic acyl group containing 6 to 22 carbon atoms, R^1 is hydrogen, an alkyl or hydroxyalkyl group containing 1 to 4 carbon atoms and [Z] is a linear or branched polyhydroxyalkyl group containing 3 to 10 carbon atoms and 3 to 10 hydroxyl groups. The polyhydroxyfatty acid amides are known substances which may normally be obtained by reductive amination of a reducing sugar with ammonia, an alkylamine or an alkanolamine and subsequent acylation with a fatty acid, a fatty acid alkyl ester or a fatty acid chloride.

The group of polyhydroxyfatty acid amides also includes compounds corresponding to formula (II):



(II)

in which R is a linear or branched alkyl or alkenyl group containing 7 to 12 carbon atoms, R^1 is a linear, branched or cyclic alkyl group or an aryl group containing 2 to 8 carbon atoms and R^2 is a linear, branched or cyclic alkyl group or an aryl group or an oxyalkyl group containing 1 to 8 carbon atoms, C_{1-4} alkyl or phenyl groups being preferred, and [Z] is a linear polyhydroxyalkyl group, of which the alkyl chain is substituted by at least two hydroxyl groups, or alkoxylated, preferably ethoxylated or propoxylated, derivatives of that group.

[Z] is preferably obtained by reductive amination of a reduced sugar, for example glucose, fructose, maltose, lactose, galactose, mannose or xylose. The N-alkoxy- or N-aryloxy-substituted compounds may then be converted into the required polyhydroxyfatty acid amides by reaction with fatty acid methyl esters in the presence of an alkoxide as catalyst, for example in accordance with the teaching of International patent application **WO-A-95/07331**.

According to the invention preferred detergent tablets contain anionic and nonionic surfactant(s). Performance-related advantages can arise out of certain quantity ratios in which the individual classes of surfactant are used.

For example, particularly preferred detergent tablets are those in which the ratio of anionic surfactant(s) to nonionic surfactant(s) is between 10:1 and 1:10, preferably between 7.5:1 and 1:5 and more preferably between 5:1 and 1:2.

Certain performance-related advantages can be obtained if certain classes of surfactant are not present in certain phases of the detergent tablets or in any of the phases. In another important embodiment of the present invention, therefore, at least one phase of the tablets is free from nonionic surfactants.

Conversely, however, a positive effect can also be obtained if individual phases or the tablet as a whole, i.e. all the phases, contain certain surfactants. The introduction of the alkyl polyglycosides described above has proved to be advantageous so that detergent tablets in which at least one phase contains alkyl polyglycosides are preferred.

As with the nonionic surfactants, the omission of anionic surfactants from individual phases or from all the phases can also result in detergent tablets which are better suited to certain applications. According to the invention, therefore, detergent tablets in which at least one phase is free from anionic surfactants are also possible.

Besides the detersive substances, builders are the most important ingredients of detergents. The detergent tablets according to the invention may contain any of the builders typically used in detergents, i.e. in particular zeolites, silicates, carbonates, organic co-builders and – providing there are no ecological objects to their use – the phosphates.

Suitable crystalline layer-form sodium silicates correspond to the general formula $NaMSi_xO_{2x+1}y\ H_2O$, 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 $Na_2Si_2O_5y\ H_2O$ are particularly preferred, β-sodium disilicate being obtainable, for example, by the process described in International patent application **WO-A- 91/08171**.

Other useful builders are amorphous sodium silicates with a modulus ($Na_2O:SiO_2$ ratio) of 1:2 to 1:3.3, preferably 1:2 to 1:2.8 and more preferably 1:2 to 1:2.6 which dissolve with delay and exhibit 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 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.

The finely crystalline, synthetic zeolite containing bound water used in accordance with the invention is preferably zeolite A and/or zeolite P. Zeolite MAP® (Crosfield) is a particularly preferred P-type zeolite. However, zeolite X and mixtures of A, X and/or P are also suitable. According to the invention, it is also possible to use, for example, a commercially obtainable 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:

$$nNa_2O \cdot (1\text{-}n)K_2O \cdot Al_2O_3 \cdot (2 - 2.5)SiO_2 \cdot (3.5 - 5.5)\ H_2O.$$

The zeolite may be used both as a builder in a granular compound and also to "powder" the entire mixture to be tabletted, both methods normally being used to incorporate the zeolite in the premix. Suitable zeolites have a mean particle size of less than 10 μm (volume distribution, as measured by the Coulter Counter Method) and contain preferably 18 to 22% by weight and more preferably 20 to 22% by weight of bound water.

The generally known phosphates may of course also be used as builders providing their use should not be avoided on ecological grounds. The sodium salts of the orthophosphates, the pyrophosphates and, in particular, the tripolyphosphates are particularly suitable.

Useful organic builders are, for example, the polycarboxylic acids usable, for example, 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), providing its 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.

In order to facilitate the disintegration of heavily compacted tablets, disintegration aids, so-called tablet disintegrators, may be incorporated in them to shorten their disintegration times. According to **Römpp (9th Edition, Vol. 6, page 4440)** and **Voigt *"Lehrbuch der pharmazeutischen Technologie"* (6th Edition, 1987, pages 182-184)**, tablet disintegrators or disintegration accelerators are auxiliaries which provide for the rapid disintegration of tablets in water or gastric juices and the release of the pharmaceuticals in an absorbable form.

These substances, which are also known as "disintegrators" by virtue of their effect, are capable of undergoing an increase in volume on contact with water so that, on the one hand, their own volume is increased

(swelling) and, on the other hand, a pressure can be generated through the release of gases which causes the tablet to disintegrate into relatively small particles. Well-known disintegrators are, for example, carbonate/citric acid systems, although other organic acids may also be used. Swelling disintegration aids are, for example, synthetic polymers, such as polyvinyl pyrrolidone (PVP), or natural polymers and modified natural substances, such as cellulose and starch and derivatives thereof, alginates or casein derivatives.

Preferred detergent tablets contain 0.5 to 10% by weight, preferably 3 to 7% by weight and more preferably 4 to 6% by weight of one or more disintegration aids, based on the weight of the tablet.

According to the invention, preferred disintegrators are cellulose-based disintegrators, so that preferred detergent tablets contain a cellulose-based disintegrator in quantities of 0.5 to 10% by weight, preferably 3 to 7% by weight and more preferably 4 to 6% by weight. Pure cellulose has the formal empirical composition $(C_6H_{10}O_5)_n$ and, formally, is a β-1,4-polyacetal of cellobiose which, in turn, is made up of two molecules of glucose. Suitable celluloses consist of ca. 500 to 5000 glucose units and, accordingly, have average molecular weights of 50,000 to 500,000. According to the invention, cellulose derivatives obtainable from cellulose by polymer-analog reactions may also be used as cellulose-based disintegrators. These chemically modified celluloses include, for example, products of esterification or etherification reactions in which hydroxy hydrogen atoms have been substituted. However, celluloses in which the hydroxy groups have been replaced by functional groups that are not attached by an oxygen atom may also be used as cellulose derivatives. The group of cellulose derivatives includes, for example, alkali metal celluloses, carboxymethyl cellulose (CMC), cellulose esters and ethers and aminocelluloses. The cellulose derivatives mentioned are preferably not

used on their own, but rather in the form of a mixture with cellulose as cellulose-based disintegrators. The content of cellulose derivatives in mixtures such as these is preferably below 50% by weight and more preferably below 20% by weight, based on the cellulose-based disintegrator. In one particularly preferred embodiment, pure cellulose free from cellulose derivatives is used as the cellulose-based disintegrator.

The cellulose used as disintegration aid is preferably not used in fine-particle form, but is converted into a coarser form, for example by granulation or compacting, before it is added to and mixed with the premixes to be tabletted. Detergent tablets which contain granular or optionally co-granulated disintegrators are described in German patent applications DE 197 09 991 (Stefan Herzog) and DE 197 10 254 (Henkel) and in International patent application **WO 98/40463** (Henkel). Further particulars of the production of granulated, compacted or co-granulated cellulose disintegrators can also be found in these patent applications. The particle sizes of such disintegration aids is mostly above 200 µm, at least 90% by weight of the particles being between 300 and 1600 µm in size and, more particularly, between 400 and 1200 µm in size. According to the invention, the above-described relatively coarse-particle cellulose-based disintegrators described in detail in the cited patent applications are preferably used as disintegration aids and are commercially obtainable, for example under the name of Arbocel® TF-30-HG from Rettenmaier.

Microcrystalline cellulose may be used as another cellulose-based disintegration aid or as part of such a component. This microcrystalline cellulose is obtained by partial hydrolysis of the celluloses under conditions which only attack and completely dissolve the amorphous regions (ca. 30% of the total cellulose mass) of the celluloses, but leave the crystalline regions (ca. 70%) undamaged. Subsequent de-aggregation of the microfine celluloses formed by hydrolysis provides the microcrystalline

celluloses which have primary particle sizes of ca. 5 µm and which can be compacted, for example, to granules with a mean particle size of 200 µm.

According to the present invention, therefore, detergent tablets additionally containing a disintegration aid, preferably a cellulose-based disintegration aid, preferably in granulated, cogranulated or compacted form, in quantities of 0.5 to 10% by weight, preferably 3 to 7% by weight and more preferably 4 to 6% by weight, based on the weight of the tablet, are particularly preferred.

Detergent tablets are produced by the application of pressure to a mixture to be tabletted which is accommodated in the cavity of a press. In the most simple method of tablet production – hereinafter referred to simply as tabletting – the mixture to be tabletted is compressed directly, i.e. without preliminary granulation. The advantages of this so-called direct tabletting are its simple and inexpensive application because no other process steps and hence no other items of equipment are involved. However, these advantages are offset by disadvantages. Thus, a powder mixture which is to be directly tabletted must possess adequate plastic deformability and good flow properties and must not show any tendency to separate during storage, transportation and filling of the die. Unfortunately, these three requirements are very difficult to satisfy with many mixtures so that direct tabletting is often not applied, particularly in the production of detergent tablets. Accordingly, the normal method of producing detergent tablets starts out from powder-form components ("primary particles") which are agglomerated or granulated by suitable methods to secondary particles with larger particle diameters. These granules or mixtures of different granules are then mixed with individual powder-form additives and the resulting mixtures are tabletted. Depending on the composition of the phases of the multiphase detergent tablets, the die is filled in steps with different premixes. In the production of multilayer tablets, the application of

light pressure between the fillings with premixes can have advantages for the next step. In the production of ring/core tablets or jacket tablets, precompression and shaping/forming such as this is even almost indispensable.

According to the invention, preferred detergent tablets are obtained by tabletting particulate premixes of at least one batch of surfactant-containing granules and at least one subsequently added powder-form component. The surfactant-containing granules may be produced by conventional granulation processes, such as mixer and pan granulation, fluidized bed granulation, extrusion, pelleting or compacting. It is of advantage so far as the subsequent detergent tablets are concerned if the premixes to be tabletted have a bulk density approaching that of standard compact detergents. In one particularly preferred embodiment, the premix to be tabletted has a bulk density of at least 500 g/l, preferably of at least 600 g/l and more preferably above 700 g/l. Another advantage can arise out of a relatively narrow particle size distribution of the surfactant granules used. According to the invention, preferred detergent tablets are those in which the granules have particle sizes of 10 to 4,000 µm, preferably between 100 and 2,000 µm and more preferably between 600 and 1,400 µm.

The invention also relates to a process for the production of two-phase or multiphase detergent tablets containing surfactant(s), builder(s) and optionally other detergent ingredients by tabletting known per se, characterized in that they are obtained by tabletting of a particulate premix of at least one batch of surfactant-containing granules and at least one subsequently incorporated powder-form component, the surfactant content of the individual phases of the tablets, based on the weight of the individual phase, varying by no more than 1.5% by weight.

So far as the variation of the surfactant content and preferred values

are concerned, the foregoing observations also apply to the process according to the invention.

Preferred processes are characterized in that the granules are produced by conventional granulation processes, such as mixer and pan granulation, fluidized bed granulation, extrusion, pelleting or compacting. In particularly preferred processes, the granules have particle sizes of 10 to 4,000 µm, preferably between 100 and 2,000 µm and more preferably between 600 and 1,400 µm.

The particle size distribution of the powder-form aftertreatment components subsequently added can also be varied, detergent tablets in which the powder-form component(s) subsequently added has/have the same particle size distribution as the granules used being preferred.

Before the particulate premix is compressed to form detergent tablets, it may be "powdered" with fine-particle surface treatment materials. This can be of advantage to the quality and physical properties of both the premix (storage, tabletting) and the final detergent tablets. Fine-particle powdering materials have been known for some time in the art, zeolites, silicates and other inorganic salts generally being used. However, the premix is preferably "powdered" with fine-particle zeolite, zeolites of the faujasite type being preferred. In the context of the present invention, the expression "zeolite of the faujasite type" encompasses all three zeolites which form the faujasite subgroup of zeolite structural group 4 (cf. Donald W. Breck: **"Zeolite Molecular Sieves"** John Wiley & Sons, New York/London/Sydney/Toronto, 1974, page 92). Besides zeolite X, therefore, zeolite Y and faujasite and mixtures of these compounds may also be used, pure zeolite X being preferred.

Mixtures or co-crystallizates of faujasite zeolites with other zeolites, which do not have to belong to zeolite structural group 4, may also be used for powdering, in which case at least 50% by weight of the powdering

rnaterial advantageously consists of a zeolite of the faujasite type.

According to the invention, preferred detergent tablets consist of a particulate premix containing granular components and subsequently incorporated powder-form components, the, or one of the, fine-particle components subsequently incorporated being a zeolite of the faujasite type with particle sizes below 100 µm, preferably below 10 µm and more preferably below 5 µm and making up at least 0.2% by weight, preferably at least 0.5% by weight and more preferably more than 1% by weight of the premix to be compressed.

The fine-particle aftertreatment components with the particle sizes mentioned above may be dry-mixed with the premix to be tabletted. However, it is also possible and preferred to "stick" them onto the surface of the relatively coarse particles by addition of small quantities of liquid components. These powdering techniques are widely described in the prior art literature and familiar to the expert. Liquid components suitable as adhesion promoters for the powdering materials are, for example, nonionic surfactants or aqueous solutions of surfactants or other detergent ingredients. In one preferred embodiment of the invention, perfume is used as the liquid component for promoting adhesion between the powdering materials and the coarse particles.

Besides the above mentioned ingredients (surfactants, builders and disintegration aids), the detergent tablets according to the invention may contain other typical detergent ingredients from the group of bleaching agents, bleach activators, enzymes, perfumes, perfume carriers, fluorescers, dyes, foam inhibitors, silicone oils, redeposition inhibitors, optical brighteners, discoloration inhibitors, dye transfer inhibitors and corrosion inhibitors.

Among the compounds yielding H_2O_2 in water which serve as bleaching agents, sodium perborate tetrahydrate and sodium perborate

monohydrate are particularly important. Other useful bleaching agents are, for example, sodium percarbonate, peroxypyrophosphates, citrate perhydrates and H_2O_2-yielding peracidic salts or peracids, such as perbenzoates, peroxophthalates, diperazelaic acid, phthaloiminoperacid or diperdodecane dioic acid.

In order to obtain an improved bleaching effect where washing is carried out at temperatures of 60°C or lower, bleach activators may be incorporated. The bleach activators may be compounds which form aliphatic peroxocarboxylic acids containing preferably 1 to 10 carbon atoms and more preferably 2 to 4 carbon atoms and/or optionally substituted perbenzoic acid under perhydrolysis conditions. Substances bearing O- and/or N-acyl groups with the number of carbon atoms mentioned and/or optionally substituted benzoyl groups are suitable. Preferred bleach activators are polyacylated alkylenediamines, more particularly tetraacetyl ethylenediamine (TAED), acylated triazine derivatives, more particularly 1,5-diacetyl-2,4-dioxohexahydro-1,3,5-triazine (DADHT), acylated glycolurils, more particularly tetraacetyl glycoluril (TAGU), N-acylimides, more particularly N-nonanoyl succinimide (NOSI), acylated phenol sulfonates, more particularly n-nonanoyl or isononanoyloxybenzene-sulfonate (n- or iso-NOBS), carboxylic anhydrides, more particularly phthalic anhydride, acylated polyhydric alcohols, more particularly triacetin, ethylene glycol diacetate and 2,5-diacetoxy-2,5-dihydrofuran.

In addition to or instead of the conventional bleach activators mentioned above, so-called bleach catalysts may also be incorporated in the tablets. Bleach catalysts are bleach-boosting transition metal salts or transition metal complexes such as, for example, manganese-, iron-, cobalt-, ruthenium- or molybdenum-salen complexes or carbonyl complexes. Manganese, iron, cobalt, ruthenium, molybdenum, titanium, vanadium and copper complexes with nitrogen-containing tripod ligands

and cobalt-, iron-, copper- and ruthenium-ammine complexes may also be used as bleach catalysts.

Suitable enzymes are those from the class of proteases, lipases, amylases, cellulases and mixtures thereof. Enzymes obtained from bacterial strains or fungi, such as Bacillus subtilis, Bacillus licheniformis and Streptomyces griseus are particularly suitable. Proteases of the subtilisin type are preferably used, proteases obtained from Bacillus lentus being particularly preferred. Of particular interest in this regard are enzyme mixtures, 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 cellulase-containing mixtures. Peroxidases or oxidases have also been successfully used in some cases. The enzymes may be adsorbed to supports and/or encapsulated in membrane materials to protect them against premature decomposition. The percentage content of enzymes, enzyme mixtures or enzyme granules in the tablets according to the invention may be, for example, about 0.1 to 5% by weight and is preferably from 0.1 to about 2% by weight.

In addition, the detergent tablets according to the invention may also contain components with a positive effect on the removability of oil and fats from textiles by washing (so-called soil repellents). This effect becomes particularly clear when a textile which has already been repeatedly washed with a detergent according to the invention containing this oil- and fat-dissolving component is soiled. Preferred oil- and fat-dissolving components include, for example, nonionic cellulose ethers, such as methyl cellulose and methyl hydroxypropyl cellulose containing 15 to 30% by weight of methoxyl groups and 1 to 15% by weight of hydroxypropoxyl groups, based on the nonionic cellulose ether, and the polymers of phthalic acid and/or terephthalic acid known from the prior art or derivatives thereof,

more particularly polymers of ethylene terephthalates and/or polyethylene glycol terephthalates or anionically and/or nonionically modified derivatives thereof. Of these, the sulfonated derivatives of phthalic acid and terephthalic acid polymers are particularly preferred.

The tablets may contain derivatives of diaminostilbenedisulfonic acid or alkali metal salts thereof as optical brighteners. Suitable optical 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 composition which contain a diethanolamino group, a methylamino group, an anilino group or a 2-methoxyethylamino group instead of the morpholino group. Brighteners of the substituted diphenyl styryl type, for example alkali metal salts of 4,4'-bis-(2-sulfostyryl)-diphenyl, 4,4'-bis-(4-chloro-3-sulfostyryl)-diphenyl or 4-(4-chlorostyryl)-4'-(2-sulfostyryl)-diphenyl, may also be present. Mixtures of the brighteners mentioned above may also be used.

Dyes and perfumes are added to the detergent tablets according to the invention to improve the aesthetic impression created by the products and to provide the consumer not only with the required washing performance but also with a visually and sensorially "typical and unmistakable" product. Suitable perfume oils or perfumes include individual perfume compounds, for example synthetic products of the ester, ether, aldehyde, ketone, alcohol and hydrocarbon type. Perfume compounds of the ester type are, for example, benzyl acetate, phenoxyethyl isobutyrate, p-tert.butyl cyclohexyl acetate, linalyl acetate, dimethyl benzyl carbinyl acetate, phenyl ethyl acetate, linalyl benzoate, benzyl formate, ethyl methyl phenyl glycinate, allyl cyclohexyl propionate, styrallyl propionate and benzyl salicylate. The ethers include, for example, benzyl ethyl ether; the aldehydes include, for example, the linear alkanals containing 8 to 18 carbon atoms, citral, citronellal, citronellyloxy-

acetaldehyde, cyclamen aldehyde, hydroxycitronellal, lilial and bourgeonal; the ketones include, for example, the ionones, α-isomethyl ionone and methyl cedryl ketone; the alcohols include anethol, citronellol, eugenol, geraniol, linalool, phenyl ethyl alcohol and terpineol and the hydrocarbons include, above all, the terpenes, such as limonene and pinene. However, mixtures of various perfumes which together produce an attractive perfume note are preferably used. Perfume oils such as these may also contain natural perfume mixtures obtainable from vegetable sources, for example pine, citrus, jasmine, patchouli, rose or ylang-ylang oil. Also suitable are clary oil, camomile oil, clove oil, melissa oil, mint oil, cinnamon leaf oil, lime blossom oil, juniper berry oil, vetiver oil, olibanum oil, galbanum oil and labdanum oil and orange blossom oil, neroli oil, orange peel oil and sandalwood oil.

The perfumes may be directly incorporated in the detergents according to the invention, although it can also be of advantage to apply the perfumes to supports which strengthen the adherence of the perfume to the washing and which provide the textiles with a long-lasting fragrance through a slower release of the perfume. Suitable support materials are, for example, cyclodextrins, the cyclodextrin/perfume complexes optionally being coated with other auxiliaries.

In order to improve their aesthetic impression, the detergent tablets according to the invention may be colored with suitable dyes. Preferred dyes, which are not difficult for the expert to choose, have high stability in storage, are not affected by the other ingredients of the detergents or by light and do not have any pronounced substantivity for textile fibers so as not to color them. Since the present invention relates to multiphase detergent tablets, considerable significance attaches to the coloring of individual phases in order to underscore the differences in active character between individual phases. Examples of the effectiveness of such coloring

and of the success of relevant claims are sufficiently known from the advertising of denture cleaning preparations.

The tablets according to the invention are produced by first dry-mixing the constituents of the individual phases, which may be completely partly pregranulated, and then forming/shaping, more particularly tabletting, the resulting mixtures using conventional processes for the production of multiphase tablets. To produce the tablets according to the invention, the premixes are compacted between two punches in a die to form a solid compactate. This process, which is referred to in short hereinafter as tabletting, comprises four phases, namely metering, compacting (elastic deformation), plastic deformation and ejection.

The tabletting process is carried out in commercially available tablet presses which, in principle, may be equipped with single or double punches. In the latter case, not only is the top punch used to build up pressure, the bottom punch also moves towards the top punch during the tabletting process while the top punch presses downwards. For small production volumes, it is preferred to use eccentric tablet presses in which the punch(es) is/are fixed to an eccentric disc which, in turn, is mounted on a shaft rotating at a certain speed. The movement of these punches is comparable with the operation of a conventional four-stroke engine. Tabletting can be carried out with a top punch and a bottom punch, although several punches can also be fixed to a single eccentric disc, in which case the number of die bores is correspondingly increased. The throughputs of eccentric presses vary according to type from a few hundred to at most 3,000 tablets per hour.

For larger throughputs, rotary tablet presses are generally used. In rotary tablet presses, a relatively large number of dies is arranged in a circle on a so-called die table. The number of dies varies – according to model – between 6 and 55, although even larger dies are commercially

available. Top and bottom punches are associated with each die on the die table, the tabletting pressures again being actively built up not only by the top punch or bottom punch, but also by both punches. The die table and the punches move about a common vertical axis, the punches being brought into the filling, compaction, plastic deformation and ejection positions by means of curved guide rails. At those places where the punches have to be raised or lowered to a particularly significant extent (filling, compaction, ejection), these curved guide rails are supported by additional push-down members, pull-down rails and ejection paths. The die is filled from a rigidly arranged feed unit, the so-called filling shoe, which is connected to a storage container for the compound. The pressure applied to the premix can be individually adjusted through the tools for the top and bottom punches, pressure being built up by the rolling of the punch shank heads past adjustable pressure rollers.

To increase throughput, rotary presses can also be equipped with two or more filling shoes. To produce two-layer or multiple-layer tablets, several filling shoes are arranged one behind the other without the lightly compacted first layer being ejected before further filling. Given suitable process control, shell and bull's-eye tablets – which have a structure resembling an onion skin – can also be produced in this way. In the case of bull's-eye tablets, the upper surface of the core or rather the core layers is not covered and thus remains visible. Rotary tablet presses can also be equipped with single or multiple punches so that, for example, an outer circle with 50 bores and an inner circle with 35 bores can be simultaneously used for tabletting. Modern rotary tablet presses have throughputs of more than one million tablets per hour.

Tabletting machines suitable for the purposes of the invention can be obtained, 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 GmbH, Berlin, Mapag Maschinenbau AG, Bern (Switzerland) and Courtoy N.V., Halle (BE/LU). One example of a particularly suitable tabletting machine is the model HPF 630 hydraulic double-pressure press manufactured by LAEIS, D.

The tablets can be made in certain shapes and certain sizes, consisting always of several phases, i.e. layers, inclusions or cores and rings. Suitable shapes are virtually any easy-to-handle shapes, for example slabs, bars, cubes, squares and corresponding shapes with flat sides and, in particular, cylindrical forms of circular or oval cross-section. This last embodiment encompasses shapes from tablets to compact cylinders with a height-to-diameter ratio of more than 1.

The portioned pressings may be formed as separate individual elements which correspond to a predetermined dose of the detergent. However, it is also possible to form pressings which combine several such units in a single pressing, smaller portioned units being easy to break off in particular through the provision of predetermined weak spots. For the use of laundry detergents in machines of the standard European type with horizontally arranged mechanics, it can be of advantage to produce the portioned pressings as cylindrical or square tablets, preferably with a diameter-to-height ratio of about 0.5:2 to 2:0.5. Commercially available hydraulic presses, eccentric presses and rotary presses are particularly suitable for the production of pressings such as these.

The three-dimensional form of another embodiment of the tablets according to the invention is adapted in its dimensions to the dispensing compartment of commercially available domestic washing machines, so that the tablets can be introduced directly, i.e. without a dosing aid, into the dispensing compartment where they dissolve on contact with water. However, it is of course readily possible to use the detergent tablets in

conjunction with a dosing aid.

Another preferred multiphase tablet which can be produced has a plate-like or slab-like structure with alternately thick long segments and thin short segments, so that individual segments can be broken off from this "multiphase bar" at the predetermined weak spots, which the short thin segments represent, and introduced into the machine. This "bar" principle can also be embodied in other geometric forms, for example vertical triangles which are only joined to one another at one of their longitudinal sides. In this case, it is appropriate for optical reasons to make the base of the triangle, by which the individual segments are interconnected, as one phase while the apex forms the second phase. In this embodiment, different coloring of the two phases is particularly attractive.

After pressing, the detergent tablets have high stability. The fracture resistance of cylindrical tablets can be determined via the diametral fracture stress. This in turn can be determined in accordance with the following equation:

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

where σ represents the diametral fracture stress (DFS) in Pa, P is the force in N which leads to the pressure applied to the tablet that results in fracture thereof, D is the diameter of the tablet in meters and t is its height.

Examples

Premixes were prepared by mixing surfactant-containing granules with powder-form aftertreatment components and were tabletted in a Korsch tablet press to form two-phase detergent tablets. Surfactant granules 1, 2 and 3 had been produced in a 130-liter plowshare mixer

(Gebrüder Lödige, Paderborn) and then dried in a fluidized-bed dryer. After the coarse fractions (≥ 1.6 mm) and the fine particles (≤ 0.4 mm) had been removed by sieving, the surfactant granules were mixed with the aftertreatment components in a paddle mixer.

The composition of the surfactant granules is shown in Table 1.

Table 1: Surfactant granules [% by weight]

	Granules 1	**Granules 2**	**Granules 3**
C_{9-13} alkyl benzenesulfonate	21.2	18.6	19.4
C_{12-18} fatty alcohol sulfate	8.5	5.4	5.2
C_{12-18} fatty alcohol + 7 EO	-	5.7	4.8
C_{12-16} alkyl-1,4-glycoside, degree of oligomerization 1.1	-	-	1.0
Soap	1.6	1.6	1.6
Sodium carbonate	17.0	16.6	17.0
Sodium silicate	5.6	5.4	5.6
Zeolite A (water-free active substance)	28.5	29.9	28.5
Optical brightener	0.3	0.3	0.3
Na hydroxyethane-1,1-diphosphate	0.8	0.8	0.8
Acrylic acid/maleic acid copolymer	5.6	5.4	5.6
Water, salts	Balance	Balance	Balance

Two-layer detergent tablets were produced from the premixes (surfactant granules + aftertreatment components) in a Korsch rotary press, the first layer making up 75% and the second layer 25% of the total weight of each tablet. The diameter of the tablets was 44 mm.

Tables 2, 3 and 4 below show the phase compositions of the

detergent tablets. The figures in the columns of the Table represent the quantity of the particular ingredient in the particular phase of the tablet, i.e. the figures in each column add up to 100%. The quantity of the particular ingredient in the tablet as a whole can easily be calculated from the percentage content of the individual phases in the tablet. Commensurate with the different tablet weights (37.5 g ± 1%,caused by slight variations in the feed of the premix to the die of the press), the tablet hardnesses varied by about ca. ± 10%, the disintegration times by ca. 5 seconds. The tablet hardnesses and disintegration times are also shown in the Tables.

Table 2: Detergent tablets – composition [% by weight], physical properties

	Example 1			
	Invention		Comparison Example	
	Layer 1	Layer 2	Layer 1	Layer 2
Granules 1	60.0	60.0	64.2	51.3
Sodium perborate monohydrate	23.7	-	23.7	-
Tetraacetyl ethylenediamine	2.6	22.5	-	29.0
Enzyme granules*	-	10.0	-	10.0
Foam inhibitor	4.7	-	3.5	1.1
Repelotex SRP 4**	1.5	-	1.1	1.1
Perfume	0.5	0.5	0.5	0.5
Zeolite A	2.0	2.0	2.0	2.0
Cellulose***	5.0	5.0	5.0	5.0
Surfactant content	18.78%	18.78%	20.09%	16.05%
Surfactant content (tablet)	18.78%		19.08%	
Tablet hardness	43-55 N		39-47 N	
Disintegration time	13-18 secs.		> 60 secs.	

* Protease, cellulase, amylase, lipase on a support (starch), coated

** Repelotex SRP 4 is a terephthalic acid/ethylene glycol, polyethylene glycol ester made by Rhône Poulenc

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

Table 3: Detergent tablets – composition [% by weight], physical properties

	Example 2			
	Invention		Comparison Example	
	Layer 1	Layer 2	Layer 1	Layer 2
Granules 2	60.0	60.1	66.8	41.1
Sodium perborate monohydrate	17.8	17.8	17.8	17.8
Tetraacetyl ethylenediamine	10.1	-	-	29.0
Enzyme granules*	-	10.0	3.3	-
Foam inhibitor	3.5	3.5	3.5	3.5
Repelotex SRP 4**	1.1	1.1	1.1	1.1
Perfume	0.5	0.5	0.5	0.5
Zeolite A	2.0	2.0	2.0	2.0
Cellulose***	5.0	5.0	5.0	5.0
Surfactant content	18.78%	18.81%	20.91%	12.86%
Surfactant content (tablet)	18.79%		18.90%	
Tablet hardness	40-50 N		37-49 N	
Disintegration time	16-22 secs.		> 60 secs.	

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

Table 4: Detergent tablets – composition [% by weight], physical properties

	Example 3			
	Invention		Comparison Example	
	Layer 1	Layer 2	Layer 1	Layer 2
Granules 3	60.1	60.1	66.3	42.5
Sodium perborate monohydrate	14.5	27.8	7.0	50.0
Tetraacetyl ethylenediamine	10.0	-	9.8	-
Enzyme granules*	3.3	-	3.3	-
Foam inhibitor	3.5	3.5	4.7	-
Repelotex SRP 4**	1.1	1.1	1.4	-
Perfume	0.5	0.5	0.5	0.5
Zeolite A	2.0	2.0	2.0	2.0
Cellulose***	5.0	5.0	5.0	5.0
Surfactant content of layer	19.23%	19.23%	21.22%	13.60%
Surfactant content (tablet)	19.23%		19.32%	
Tablet hardness	43-51 N		38-47 N	
Disintegration time	13-17 secs.		> 60 secs.	

* Protease, cellulase, amylase, lipase on a support (starch), coated

** Repelotex SRP 4 is a terephthalic acid/ethylene glycol, polyethylene glycol ester made by Rhône Poulenc

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

CLAIMS

1. Two-phase or multiphase detergent tablets of compacted particulate detergent comprising surfactants, builders and optionally other detergent ingredients, wherein the surfactant content of the individual phases of the tablets varies by no more than 1.5% by weight, based on the weight of the individual phase.

2. Detergent tablets as claimed in claim 1, wherein the surfactant content of the individual phases differs by no more than 1% by weight, based on the weight of the individual phase.

3. Detergent tablets as claimed in claim 1 or 2, wherein the surfactant content of the individual phases of the tablet is identical.

4. Detergent tablets as claimed in any of claims 1 to 3, wherein the phases of the tablets are in the form of layers.

5. Detergent tablets as claimed in any of claims 1 to 4, wherein the surfactant(s) are introduced into the phases of the tablets through one or more batches of surfactant-containing granules.

6. Detergent tablets as claimed in claim 5, wherein the same surfactant granules are used in all phases of the tablets.

7. Detergent tablets as claimed in claims 4 and 6, wherein there are two layers which contain the same surfactant granules.

8. Detergent tablets as claimed in any of claims 1 to 7, wherein anionic and nonionic surfactants are present.

9. Detergent tablets as claimed in claim 8, wherein the ratio of anionic surfactants to nonionic surfactants is between 10:1 and 1:10.

10. Detergent tablets as claimed in claim 8, wherein the ratio of anionic surfactants to nonionic surfactants is between 7.5:1 and 1:5.

11. Detergent tablets as claimed in claim 8, wherein the ratio of anionic surfactants to nonionic surfactants is between 5:1 and 1:2.

12. Detergent tablets as claimed in any of claims 1 to 6, wherein at least

one phase of the tablets is free from nonionic surfactants.

13. Detergent tablets as claimed in any of claims 1 to 6, wherein at least one phase of the tablets contains alkyl polyglycosides.

14. Detergent tablets as claimed in any of claims 1 to 13, wherein at least one phase of the tablets is free from anionic surfactants.

15. Detergent tablets as claimed in any of claims 1 to 14, wherein there is additionally present a disintegration aid, in quantities of 0.5 to 10% by weight, based on the weight of the tablet.

16. Detergent tablets as claimed in claim 15 wherein the disintegration aid is a cellulose-based disintegration aid.

17. Detergent tablets as claimed in claim 15 or 16 wherein the disintegration aid is in granulated, cogranulated or compacted form.

18. Detergent tablets as claimed in any of claims 14 to 17, wherein the disintegration aid is present in quantities of 3 to 7% by weight, based on the weight of the tablet.

19. Detergent tablets as claimed in any of claims 14 to 17, wherein the disintegration aid is present in quantities of 4 to 6% by weight, based on the weight of the tablet.

20. Detergent tablets as claimed in any of claims 1 to 19 wherein there is additionally present one or more substances from the group of builders, bleaching agents, bleach activators, enzymes, pH regulators, perfumes, perfume carriers, fluorescers, dyes, foam inhibitors, silicone oils, redeposition inhibitors, optical brighteners, discoloration inhibitors, dye transfer inhibitors and corrosion inhibitors.

21. A process for the production of two-phase or multiphase detergent tablets containing surfactant(s), builder(s) and optionally other detergent ingredients by tabletting known per se, wherein they are obtained by tabletting of a particulate premix of at least one batch of surfactant-containing granules and at least one subsequently incorporated powder-

form component, the surfactant content of the individual phases of the tablets, based on the weight of the individual phase, varying by no more than 1.5% by weight.

22. A process as claimed in claim 21, wherein the granules are produced by a granulation process selected from mixer and pan granulation, fluidized bed granulation, extrusion, pelleting or compacting.

23. A process as claimed in claim 21 or 22, wherein the granules have particle sizes of 10 to 4,000 µm.

24. A process as claimed in claim 21 or 22, wherein the granules have particle sizes of between 100 and 2,000 µm.

25. A process as claimed in claim 21 or 22, wherein the granules have particle sizes of between 600 and 1,400 µm.

26. A process as claimed in any of claims 21 to 25, wherein the powder-form component(s) subsequently incorporated have the same particle size distribution as the granules used.

27. A process as claimed in any of claims 21 to 26, wherein the premix to be tabletted has a bulk density of at least 500 g/l.

28. A process as claimed in any of claims 21 to 27, wherein the premix to be tabletted has a bulk density of at least 600 g/l.

29. A process as claimed in any of claims 21 to 28, wherein the premix to be tabletted has a bulk density above 700 g/l.

30. A process as claimed in any of claims 21 to 29, wherein one of the powder-form components subsequently incorporated is a faujasite zeolite with particle sizes below 100 µm and makes up at least 0.2% by weight of the premix to be tabletted.

31. A process as claimed in claim 30 wherein one of the powder-form components subsequently incorporated is a faujasite zeolite with particle sizes below 10 µm.

32. A process as claimed in claim 30 wherein one of the powder-form

components subsequently incorporated is a faujasite zeolite with particle sizes below 5 µm.

33. A process as claimed in claim 30 to 32 wherein one of the powder-form components subsequently incorporated is a faujasite zeolite and makes up at least 0.5% by weight of the premix to be tabletted.

34. A process as claimed in claim 30 to 32 wherein one of the powder-form components subsequently incorporated is a faujasite zeolite and makes up at least 1% by weight of the premix to be tabletted.