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(54) High bulk density granular detergent compositions and process for preparing them.

(57) There is provided a granular detergent composition or component having a bulk density of at least 600 g/land good dispensing properties, and comprising from 10 to 70% by weight of a builder, at least 50% by weight of said builder being a non-phosphate material, and from 5 to 45% by weight of a ternary active system comprising one or more nonionic surfactants, anionic surfactants and soap, whereby the weight ratio of the anionic surfactant to the nonionic surfactant is less than 5 : 1 and the amount of soap is from 10 to 90% by weight of the active system. There is also provided a process for preparing such compositions.

TECHNICAL FIELD

The present invention relates to granular detergent compositions having a high bulk density and good dispensing properties. Moreover, it relates to a process for the preparation of such detergent compositions, and more in particular, to a process for their continuous preparation.

BACKGROUND AND PRIOR ART

Recently there has been considerable interest within the detergents industry in detergent powders having relatively high bulk density, for example 600 g/litre and above.

There are several processes available in the art by which densified detergent powders may be made. Particular attention has thereby been paid to the densification of spray-dried powders by post-tower treatment. For instance, JP-A-61 069897 (KAO) discloses a process in which a spray-dried detergent powder containing a high level of anionic surfactant and a low level of builder (zeolite) is subjected successively to pulverizing and granulating treatments in a high-speed mixer/granulator, the granulation being carried out in the presence of an "agent for improving surface properties" and optionally a binder. It would appear that in the high-speed mixer/granulator, the spray-dried powder is initially broken down to a fine state of division; the surface-improving agent and optional binder are then added and the pulverized material granulated to form a final product of high bulk density. The surface-improving agent, which is a finely divided particulate solid such as fine sodium aluminosilicate, is apparently required in order to prevent the composition from being formed into large balls or cakes. The process described in this Japanese patent application is essentially a batch process and is therefore less suitable for the large scale production of detergent powders.

GB-A-1 517 713 (UNILEVER) discloses a batch process in which spray-dried or granulated detergent powders containing sodium tripolyphosphate and sodium sulphate are densified and spheronized in a "marumerizer" (Trade Mark). This apparatus comprises a substantially horizontal, roughened, rotatable table positioned within, and at the base of, a substantially vertical, smooth-walled cylinder.

GB-A-1 453 697 (UNILEVER) discloses the use of a "marumarizer" (Trade Mark) for granulating together detergent powder components in the presence of a liquid binder to form a granular detergent composition. The disadvantage associated with this apparatus is that it produces powders or granules having a rather wide particle size distribution, and in particular containing a relatively high proportion of oversize particles. Such products exhibit poor dissolution and dispersion characteristics, particularly in low-temperature short duration machine washes as used in Japanese and other far-eastern washing machines. This can be apparent to the consumer as deposits on washed fabrics, and in machine washing leads to a high level of wastage.

EP-A-327 963 (HENKEL) describes a continuous process for increasing the bulk density of a spray-dried detergent powder by treating it in a mixing apparatus consisting of a substantially horizontal static hollow cylinder and a rotating shaft in the middle, the shaft having several different types of blades mounted thereon. Example 1 discloses a densified detergent powder having a bulk density of 595 g/l, containing a ternary active system consisting of an anionic surfactant, nonionic surfactants and soap, the amount of soap being about 13% of the active system. The formulation further contains a builder system consisting of 10% zeolite and 20% sodium tripolyphosphate, whereby the latter is likely to account for the claimed favorable dispensing properties.

The related EP-A-337 330 (HENKEL) describes a variant of the previous process, whereby liquid nonionic surfactant is sprayed onto a spray-dried base powder. The base powder is a low-phosphate base powder and comprises conventional ingredients in the usual amounts. The process results in a moderate increase in bulk density of at most 100 g/l.

EP-A-220 024 (Procter & Gamble) discloses a process in which a spray-dried detergent powder containing a high level (30-85% by weight) of anionic surfactant is mixed with an inorganic builder (sodium tripolyphosphate, or sodium aluminosilicate and sodium carbonate) and compacted under high pressure using a roll compactor ("chilsonator"); the compacted material, after removal of oversize material and fines, is then granulated using conventional apparatus, for example a fluidized bed, tumble mixer, or rotating drum or pan.

Although it is possible by means of the above-mentioned processes to prepare detergent powders having an increased bulk density, the obtained powders all have the draw-back that they dispense less well in European-type automatic washing machines than the corresponding undensified powders. As a consequence, a higher proportion of the powder dosed into the machine is left behind in the dispenser, leading to powder wastage and clogging. This problem is especially marked with detergent powders containing little or no tripolyphosphate, and at low wash temperatures.

As a consequence of the poor dispensing characteristics of the known densified detergent powders, it is necessary to use them in combination with a dispensing device or shuttle. This restricts the method of use of the product, which is not in all cases favorably perceived by the consumer, and the shuttles which are usually made of a plastic material may contribute to waste-disposal problems.

It is therefore an object of the present invention to provide high bulk density granular detergent compositions or components thereof, having a bulk density of at least 600 g/l, preferably at least 650 g/l, and which nevertheless possess good dispensing characteristics. It is another object of the invention to provide a process for obtaining such compositions. The process should be especially suitable for the large scale manufacture of such compositions, and should therefore preferably be a continuous process.

We have now found that granular detergent compositions having a low phosphate content and a bulk density of at least 600 g/l, and yet having surprisingly good dispensing properties may be obtained if certain requirements with regard to the formulation are observed.

DEFINITION OF THE INVENTION

In a first aspect, the present invention provides a granular detergent composition or component having a bulk density of at least 600 g/l, comprising from 10 to 70% by weight of a builder, at least 50% by weight of said builder being a non-phosphate material, and from 5 to 45% by weight of a ternary active system comprising one or more nonionic surfactants, anionic surfactants and soap, whereby the weight ratio of the anionic surfactant to the nonionic surfactant is less than 5 : 1 and the amount of soap is from 10 to 90% by weight of the active system.

In a second aspect, the invention provides a process for the preparation of the granular detergent composition or component of the invention, whereby a particulate starting material is treated

(i) in a high-speed mixer/densifier, the mean residence time being from about 5-30 seconds; and then
(ii) in a drying and/or cooling apparatus.

Preferably, the particulate starting material is brought into, or maintained in, a deformable state in the first step.

DETAILED DESCRIPTION OF THE INVENTION

The granular detergent compositions according to the invention comprise 10 to 70% by weight of a builder system and 5 to 45% by weight of an active system.

The builder system of the compositions according to the invention may consist of a single detergency builder in a amount of from 10 to 70 % by weight of the total formulation. It may also be a mixture of two or more detergency builders. However, the invention is especially applicable for detergent powders wherein at least 50% by weight of the builder system is a non-phosphate material, because in such cases the dispensing properties of densified powders of conventional formulations are particularly poor.

The builder may be any material capable of reducing the level of free calcium ions in the wash liquor and will preferably provide the composition with other beneficial properties such as the generation of an alkaline pH, the suspension of soil removed from the fabric and the suspension of the fabric-softening clay material. The level of the detergency builder is preferably from 15% to 60% by weight.

Examples of suitable detergency builders which may be used in the invention include precipitating builders such as the alkali metal carbonates, bicarbonates, orthophosphates, sequestering builders such as the alkali metal tripolyphosphates or nitrilotriacetates, or ion exchange builders such as the amorphous alkali metal aluminosilicates or the zeolites, or layered silicates, for example Na-SKS-6 from Hoechst. Preferably, the detergency builder is a non-phosphate builder such as zeolite.

The active system of the compositions according to the invention is a ternary detergent active system, consisting of an anionic surfactant, a nonionic surfactant and a soap. It is present in an amount of from 5 to 45 % by weight of the total formulation.

It was found to be essential that the weight ratio of the anionic to the nonionic surfactant is less than 5 : 1, preferably less than 4 : 1. Furthermore, the amount of soap must at least be 10 % by weight and less than 90% by weight of the active system, in order to obtain the desired good dispensing properties. Preferably, the amount of soap is from 10 to 60% by weight of the active system.

The anionic surfactants of the ternary active system are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher (C_8-C_{18}) alcohols, produced for example from tallow or coconut oil, sodium and potassium alkyl (C_9-C_{20}) benzene sulphonates, particularly sodium linear secondary alkyl (C_{10}-C_{15}) benzene sulphonates; and sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum. The preferred anionic detergent compounds are sodium (C_{11}-C_{15}) alkyl benzene sulphonates and sodium (C_{16}-C_{18}) alkyl sulphates.

Suitable nonionic detergent compounds which may be used in the ternary active system include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl (C_6-C_{22}) phenols-ethylene oxide condensates, generally 3 to 25 EO, i.e. 3 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic (C_8-C_{18}) primary or secondary linear or branched alcohols with ethylene oxide, averaging generally from 3 to 40 EO. Alkoxylated fatty alcohols, more in particular ethoxylated alcohols are the preferred nonionic surfactants.

The soaps which are used in the compositions of the present invention are sodium salts of fatty acids of natural or synthetic origin. The alkyl groups of the fatty acids may be branched or straight alkyl groups comprising 8 to 22 carbon atoms, preferably from 12 to 20 carbon atoms.

Particularly referred ternary active systems are mixtures of a sodium salt of an alkyl benzene sulphonate, an ethoxylated alcohol and a sodium soap having 12-20 carbon atoms.

Minor amounts of amphoteric or zwitterionic detergent compounds can also be used in the compositions of the invention but this in not normally desired owing to their relatively high cost.

The detergent powder of the invention may contain any of the ingredients conventionally present in compositions intended for the washing of fabrics. If desired, the powder of the invention may contain sodium silicate. High levels of silicate can in themselves have a beneficial effect on dispensing, as well as on powder structure and prevention of machine corrosion, but are undesirable in powders containing aluminosilicate because the two components react together to form insoluble siliceous species. Accordingly, the invention is of especial applicability to powders containing less that 5% by weight, more especially less than 2% by weight, of sodium silicate, which would be expected to have poor dispensing properties.

The granular detergent composition or component of the invention may be used as a detergent powder of its own right, but it can also be used as a base powder for formulating a complete fabric washing powder by adding other components. Examples of such components include inorganic salts such a sodium carbonate, sodium silicate, etc., bleaches, fluorescers, lather control agents, enzymes and perfumes. The final product will usually comprise from 50 to 95% by weight of the base powder described above.

The addition of more dense materials such as perborate and/or materials having a small particle size may even further increase the bulk density to values of up to 700 g/l and above.

The granular detergent composition or component of the invention may be prepared by any suitable technique, whether batch or continuous, for obtaining detergent compositions having an increased bulk density of 600 g/l and above. According to a preferred process, a particulate starting material is treated

(i) in a high-speed mixer/densifier, the mean residence time being from about 5-30 seconds; and then

(ii) in a drying and/or cooling apparatus.

In the first step of this process, a particulate starting material is thoroughly mixed in a high-speed mixer/densifier for a relatively short time of about 5-30 seconds.

The particulate starting material may be prepared by any suitable method, such as spray-drying or dry-mixing. The process is therefore very flexible with respect to the chemical composition of the starting material. Phosphate-containing as well as zeolite-containing compositions, and compositions having either a low or a high active content may be used. The process is also suitable for densifying calcite/carbonate-containing detergent compositions.

If a spray-dried powder is used as the particulate starting material, the particle porosity is considerable and a large increase in bulk density can be obtained by the process of this invention.

For obtaining an optimal densification it was found to be important to subject the particulate starting material to a two-step densification process. The first step is carried out in a high-speed mixer/densifier, preferably under conditions whereby the starting material is brought into, or maintained in, a deformable state, to be defined hereafter. As a high-speed mixer/densifier we advantageously used the Lödige (Trade Mark) CB 30 or CB 100 Recycler. These apparatus essentially consist of a large static hollow cylinder and a rotating shaft in the middle. The shaft has several different types of blades mounted thereon. It can be rotated at speeds between 100 and 2500 rpm, dependent on the degree of densification and the particle size desired. The blades on the shaft provide a thorough mixing action of the solids and the liquids which may be admixed in this stage. The mean residence time is somewhat dependent on the rotational speed of the shaft, the position of the blades and the weir at the exit opening. It is also possible to add solid material in the Lödige recycler.

Other types of high-speed mixers/densifiers having a comparable effect on detergent powders can also be contemplated. For instance, a Shugi (Trade Mark) Granulator or a Drais (Trade Mark) K-TTP 80 could be used.

For use, handling and storage, the detergent powder must obviously no longer be in a deformable state. Therefore, in a last processing step according to the present invention, the densified powder is dried and/or cooled. This step can be carried out in a known way, for instance in a fluid bed apparatus (drying) or in an airlift (cooling). From a processing point of view, it is advantageous if the powder needs a cooling step only, because the required equipment is relatively simple.

After the first step of the process of the invention, the detergent material may still possess a considerable porosity such that a further increase in bulk density could be achieved. Instead of choosing a longer residence time in the high-speed mixer/densifier to try to obtain a further bulk density increase, we found it to be particularly advantageous to subject the powder to a further densification step. The process is then essentially the same as described in our co-pending not prepublished European patent application 367,339.

In this further processing step the detergent material is treated for 1-10 minutes, preferably for 2-5 minutes, in a moderate-speed granulator/densifier under such conditions that the powder is brought into, or maintained in, a deformable state. As a consequence, the particle porosity will be further reduced. The main differences with the first step reside in the lower mixing speed and the longer residence time of 1-10 minutes.

This further processing step can be successfully carried out in a Lödige (Trade Mark) KM 300 mixer, also referred to as Lödige Ploughshare. This apparatus essentially consists of a hollow static cylinder having a rotating shaft in the middle. On this shaft various plough-shaped blades are mounted. It can be rotated at a speed of 40-160 rpm. Optionally, one or more high-speed cutters can be used to prevent excessive agglomeration. Another suitable machine for this step is, for example, the Drais (Trade Mark) K-T 160.

Optionally, small amounts of fine powdered solids, such as zeolite, can be added in the high-speed mixer/densifier and/or the moderate speed granulator/densifier, such as disclosed in our co-pending European patent application 390,251.

Preferred for the first step and essential for the further processing step is the deformable state into which the detergent powder must be brought in order to get optimal densification. This deformable state may be induced in a number of ways, for instance by operating at temperatures above 45 ° C. When liquids such as water or nonionics are added to the particulate starting material, lower temperatures may be employed, for example 35 ° C and above.

According to a preferred embodiment of the present invention, a spray-dried base powder leaving the tower at a temperature of above 45 ° C is fed directly into the process of the present invention.

Alternatively, the spray-dried powder may be cooled first, e.g. in an airlift, and subsequently be heated again after transportation. The heat may be applied externally, possibly supplemented by internally generated heat, such as heat of hydration of water-free sodium tripolyphosphate.

The deformability of a detergent powder can be derived from its compression modulus, which in turn can be derived from its stress-strain characteristics. To determine the compression modulus of a specific composition and moisture content, a sample of the composition is compressed to form an airless prill of 13 mm diameter and height. Using an Instron testing machine, the stress-strain diagram during unconfined compression is recorded at a constant strain rate of 10 mm/min. The compression modulus can now be derived from the slope of the stress - versus relative strain diagram during the first part of the compression process, which reflects the elastic deformation. The compression modulus is expressed in MPa (mega Pascal). In order to measure the compression modulus at various temperatures, the Instron apparatus can be equipped with a heatable sample holder.

The compression modulus as measured according to the above method was found to correlate well with the particle porosity decrease and the accompanying bulk density increase, under comparable processing conditions. This is further illustrated in the Examples.

As a general rule, the powder can be considered in a deformable state if the compression modulus as defined above is less than approximately 25, preferably less than 20 MPa. Even more preferably, the compression modulus is less than 15 MPa and values of less than 10 MPa are particularly preferred.

The deformability of a powder depends, among other things, on the chemical composition, the temperature and the moisture content. As to the chemical composition, the liquids to solids ratio and the amount of polymer proved to be important factors. Moreover, it was generally more difficult to bring phosphate-containing powders into a deformable state than it was for zeolite-containing powders.

The storage stability of the final detergent powder can be assessed by means of the Unconfined Compressibility Test. In this test the detergent powder is placed in a cylinder having a diameter of 13 cm and a height of 15 cm. Subsequently, a weight of 10 kg is placed on top of the powder. After 5 minutes the weight is removed and the walls of the cylinder are taken away. Then an increasing load is placed on top of the column of compressed detergent powder and the weight (in kg) is determined at which the column disintegrates. This value is a function of the stickiness of the detergent powder and proved to be a good measure for the storage stability.

Dispensing performance is assesssed by the following method. Dry powder (100 g) is placed in a dry dispenser tray of an automatic front-loading washing machine of the type Philips AWB 126/127 (shower-type dispenser). Mains water at a flow rate of 5 liters/min, supplied at a pressure of 0.5 bar, is allowed to flow through the dispenser for one minute. The temperature of the water is 10-20 °C. The remaining undispensed powder is removed, dried at 100 °C for 12 hours, and weighed. The dispensing residue is the residual dried powder expressed as a percentage of the initial sample. An average of 4 determinations is taken as the final result.

The invention will now be further illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated. In the Examples, the following abbreviations are used:

LAS	: C_{12}-C_{15} Linear alkyl benzene sulphonate
Nonionic	: Ethoxylated C_{12}-C_{15} fatty alcohol nonionic surfactant
Soap	: sodium salt of C_{15}-C_{20} fatty acid
Zeolite	: Zeolite 4A (Wessalith [Trade Mark] ex Degussa)
Carbonate	: Sodium carbonate
Sulphate	: Sodium sulphate
Silicate	: Sodium alkaline silicate; Na_2O:SiO_2 ratio 1:2
Polymer	: Copolymer of maleic and acrylic acid having a molecular weight of 70,000; CP5 ex BASF
Antifoam	: Silicone oil antifoam granule

EXAMPLES 1-6

Detergent powders were prepared to the compositions given in Table 1 by spray-drying aqueous slurries. Examples 1-3 relate to products produced within the scope of the invention, the examples 4 and 5 are comparative Examples. The amounts are parts by weight.

TABLE 1

Example	1	2	3	4	5
LAS	6.4	8.0	8.4	9.0	7.2
Nonionic.6-8 EO	3.2	1.0	3.9	1.0	3.3
Soap	1.9	1.8	2.4	0.5	2.0
Zeolite	25.5	24.0	24.0	24.0	29.0
Polymer	4.3	4.0	3.0	4.0	5.0
Carbonate	9.0	2.0	8.5	2.0	10.0
Silicate	0.5	0.5	nil	0.2	0.5
Minor ingredients	1.2	1.7	1.0	2.0	1.6
Water	9.6	10.0	10.8	8.5	12.5

The powders were produced in a pilot plant at 0.5-1.0 t/hour or at full scale at 20-30 tonnes per hour. The temperature at the tower base was about 60-70 °C. The physical properties of the spray-dried powders are given in Table 2.

TABLE 2

Example	1	2	3	4	5
Bulk density (g/l)	440	444	443	474	458
Moisture content (%) #	14.5	13.3	12.2	11.1	13.9
Mean particle size (microns) *	646	758	682	527	590

by Infra-red moisture balance at 135°C

* using Rosin-Rammler system; size of theoretical sieve that retains 37% of powder.

The powders were fed directly into a Loedige Recycler CB continuous high speed mixer/densifier, described in more detail above. The CB 30 unit was used for the low throughput described, and the CB 100 for the high throughput. The units were usually operated at a mixer tip speed of 8-30 meters per second. The comparative powder of example 5 was treated in a conventional rotating drum mixer. The residence time was in this case about 1 to 2 minutes.

In the Recycler and the drum, various solids and/or liquids and/or binders were added, as indicated in Table 3. The properties of the powders after leaving the Loedige Recycler or rotating drum are given also in Table 3.

TABLE 3

Example	1a	1b	2	3	4	5
Densifications:						
Throughput ton/hr	23	0.8	0.8	0.9	0.8	23
Type of Mixer	CB	CB	CB	CB	CB	drum
Mixer speed (rpm)	150-250	600-1000	1500	1800	1000	12
Addition of:						
Nonionic.6-7 EO	0	2	3	0	0	0
Nonionic.3EO	4-7	2	5	3.7	3	7
Silicate 33% soln.	0	3	0	0	0	0
Carbonate	0	0	0	3-5	0	0
Bulk density (kg/m3)	635	642	613	647	620	560
Moisture content (%) #	12.3	14.2	12.7	10.5	12.1	13.7
Mean Particle size (um) *	526	560	590	742	510	610

The powders can be optionally passed through an additional moderate speed granulator/densifier. This was done with the powder of Example 3, to give the properties shown in Table 4.

TABLE 4

Bulk density (kg/m3)	859
Mean particle size (um) *	671

To obtain a final base powder, a cooling/drying step was carried out in a fluid bed. This yielded base powder with the properties shown in Table 5.

TABLE 5

Example	1a	1b	2	3	4	5
Bulk density (g/l)	652	633	638	790	627	585
Particle size (um)*	490	540	585	690	497	592
Moisture content (%) #	12.3	12.6	11.5	9.8	11.9	13.2

Finally, the base powders were supplemented with bleach, enzymes, antifoam granules (optionally containing silicone oil) perfume etc. Details of added ingredients, final powder properties, and dispensing behaviour are given in Table 6.

TABLE 6

Example	1a	1b	2	3	4	5
Added ingredients:						
Perborate	16.5	8	8	16.3	8	14
TAED	6.0	3	3	6.6	2.8	6
Antifoam	1.5	2	2.5	2.0	0	1.2
Carbonate	4.5	0	0	0	9	0
Sulphate	0	29	8	0	12.5	0
Coloured speckles	0.3	0	0	0.5	0	0.5
Perfume	0.6	0	0	0.6	0.3	0.2
Enzymes	1.2	0.5	0.5	0.6	0.8	0.9
Bulkdensity (g/l)	654-692	779	682	768	737-780	595
Dynamic flow rate (ml/s)	108	100	82	93	89-121	110
Mean Particle size (um) *	470-550	453	513	638	375-592	380
Dispensing residue (%) (at 10-20°C)	nil	nil	0-5	0-5	40-60	nil

The good dispensing properties of the compositions according to the invention are clearly evident by comparison of examples 1-3 with example 4. Example 4 which lies outside the required detergent active specification because the soap content of the active system is too low, exhibits poor dispensing properties.

The advantages of the process of the present invention are clearly illustrated by comparison of Examples 1-3 with Example 5. The powder of example 5 was produced in a conventional drum without the densification step and has a bulk density of only 595 g/l.

Claims

1. Granular detergent composition or component having a bulk density of at least 600 g/l, comprising from 10 to 70% by weight of a builder, at least 50% by weight of said builder being a non-phosphate material, and from 5 to 45% by weight of a ternary active system comprising one or more nonionic surfactants, anionic surfactants and soap, whereby the weight ratio of the anionic surfactant to the nonionic surfactant is less than 5 : 1 and the amount of soap is from 10 to 90% by weight of the active system.

2. Granular detergent composition or component according to Claim 1, whereby the weight ratio of the anionic surfactant to the nonionic surfactant is less than 4 : 1, and the amount of soap is from 10 to 60% by weight of the active system.

3. Granular detergent composition or component according to any one of the preceding Claims, having a bulk density of at least 650 g/l.

4. Granular detergent composition or component according to any one of the preceding Claims wherein the builder is a non-phosphate builder.

5. Granular detergent composition or component according to Claim 4, wherein the builder is zeolite.

6. Granular detergent composition or component according to any one of the preceding Claims, containing less than 5, preferably less than 2% by weight of silicate.

7. Granular detergent composition or component according to any one of the preceding Claims, being substantially free of alkali metal sulphates.

8. Detergent powder comprising 50 to 95% by weight of the granular detergent composition or component according to any one of the preceding Claims.

9. Process for the preparation of a granular detergent composition or component according to Claims 1-7, whereby a particulate starting material is treated
   (i) in a high-speed mixer/densifier, the mean residence time being from about 5-30 seconds; and then
   (ii) in a drying and/or cooling apparatus.

10. Process according to Claim 9, wherein the particulate starting material is brought into, or maintained in, a deformable state as herein defined.

11. Process according to Claims 9-10, wherein the deformable state is brought about by operating at temperatures above 45°C and/or adding liquid to the particulate starting material.

12. Process according to Claims 9-11, wherein nonionics and/or water are sprayed on to the particulate starting material.

13. Process according to Claims 9-12, wherein the particulate starting material is a spray-dried detergents base powder.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91200662.4

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	<u>EP - A1 - 0 339 996</u> (UNILEVER NV) * Claims 1-5; examples * --	1-13	C 11 D 10/04 C 11 D 11/00 C 11 D 17/06
D,X	<u>EP - A2 - 0 327 963</u> (HENKEL KGaA) * Claim 1; examples; page 4, line 58 - page 5, lines 10, 27-29 * --	1-6, 8-11, 13	
Y	<u>AT - B - 337 325</u> (HENKEL & CIE. GMBH) * Examples 3,5,6,8,10 * --	1-7	
D,Y	<u>GB - A - 1 517 713</u> (UNILEVER LTD.) * Examples 2,7 * --	1-7	
A	<u>EP - A2 - 0 150 613</u> (UNILEVER NV) * Page 20 * ----	1,2,4, 5	**TECHNICAL FIELDS SEARCHED (Int. Cl.5)**
			C 11 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	07-06-1991	REISER

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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