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(54) Detergent compositions and process for preparing them.

(57) A granular detergent composition or component therefor, which is preferably free of phosphate builders, has a bulk density of at least 650 g/litre and comprises from 17 to 35 wt% of non-soap detergent-active material (consisting at least partially of anionic detergent-active material) and from 28 to 45 wt% (anhydrous basis) of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being from 0.9:1 to 2.6:1. The particle porosity is preferably less than 0.20. The composition may be prepared by granulation and densification in a high-speed mixer/granulator.

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Description

DETERGENT COMPOSITIONS AND PROCESS FOR PREPARING THEM

TECHNICAL FIELD

The present invention relates to granular detergent compositions of high bulk density having good washing performance and good powder properties, and a process for preparing them.

BACKGROUND AND PRIOR ART

Recently there has been considerable interest within the detergents industry in the production of detergent powders having relatively high bulk density, for example, 600 g/litre and above. Particular attention has been paid to the densification of spray-dried powders by post-treatment. EP 219 328A (Unilever) discloses a granular low-phosphate detergent composition prepared by spray-drying a slurry to give a base powder containing a low to moderate level of sodium tripolyphosphate builder and low levels of inorganic salts, and then postdosing solid material including sodium sulphate of high bulk density and of smaller particle size than the base powder, thus filling the voids between base powder particles and producing a product of high bulk density. EP 270 240A (Unilever), published on 8 June 1988, discloses a spray-dried zero-phosphate base powder having a particle porosity of less than 0.40 and containing anionic surfactant, optional nonionic surfactant, aluminosilicate builder, polymeric polycarboxylate and a low or zero level of electrolyte, preferably sodium carbonate. If desired, sodium sulphate or other solids of small particle size and high bulk density may be postdosed to the spray-dried powder to give compositions of very high bulk density.

EP 229 671A (Kao) discloses postdosing a crystalline alkaline inorganic salt, for example, sodium carbonate, to a spray-dried base powder to produce a high bulk density product.

A different approach is described in GB 1 517 713 (Unilever), which discloses a process in which a detergent powder produced by spray-drying or pan granulation is spheronised and granulated in a "marumerizer" (Trade Mark) with some increase in bulk density.

JP 61 069897A (Kao) discloses a process in which a spray-dried detergent powder containing surfactant and builder is subjected successively to pulverising 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 pulverised 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 forming into large balls or cakes: the examples of spray-dried starting powders described in the Kao specification contain very high levels of surfactant (45 wt%) and relatively low levels of builder salts, and are likely to have a high tendency towards caking and balling.

EP 220 024A (Procter & Gamble) is also concerned with the densification of a spray-dried powder containing a high level (30-85 wt%) of anionic surfactant. The powder is compacted and granulated, inorganic builder (sodium tripolyphosphate, or sodium aluminosilicate and sodium carbonate) being added before compaction.

The present inventors have now discovered that detergent compositions of high bulk density can be prepared by granulating a spray-dried or dry-mixed powder in a high-speed mixer/granulator, if necessary after pulverisation, without the need for an "agent for improving surface properties" or similar pulverulent material, if the initial powder is correctly formulated, especially with regard to the amount of surfactant and the ratio of surfactant to builder present.

DEFINITION OF THE INVENTION

In a first aspect, the present invention provides a granular detergent composition or component therefor having a bulk density of at least 650 g/litre, which comprises:

(a) from 17 to 35 wt% of non-soap detergent-active material consisting at least partially of anionic detergent-active material;

(b) from 28 to 45 wt% (anhydrous basis) of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being from 0.9:1 to 2.6:1, preferably from 1.2:1 to 1.8:1; and optionally other detergent ingredients to 100 wt%.

In a second aspect, the present invention provides a process for the preparation of a granular detergent composition or component having a bulk density of at least 650 g/litre, which comprises the step of treating a particulate starting material comprising:

(a) from 5 to 35 wt% of non-soap detergent-active material consisting at least partially of anionic detergent-active material, and

(b) from 28 to 45 wt% (anhydrous basis) of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being at least 0.9:1, and optionally other detergent components to 100 wt%,

in a high-speed mixer/granulator having both a stirring action and a cutting action, in the presence of a liquid binder but in the absence of a finely divided particulate agent for improving surface properties, whereby granulation and densification to a bulk density of at least 650 g/litre are effected.

DETAILED DESCRIPTION OF THE INVENTION

The product

The first aspect of the invention is a dense granular detergent powder combining high bulk density, good powder properties and excellent washing and cleaning performance, that can be prepared easily and conveniently by the process that is also the subject of the present invention.

The detergent composition of the invention owes its combination of excellent properties and ready processability to a moderate content of surfactant, at least part of which is anionic, and a relatively high level of sodium aluminosilicate builder. The present inventors have found that when the absolute amounts of aluminosilicate builder and surfactant in a powder, and the ratio of one to the other, are suitably chosen, that powder may be granulated in a high-speed mixer/granulator without the need for the use of an "agent for improving surface properties" during the granulation step as prescribed by JP 61 069897A (Kao). The resulting dense granulate has good flow properties and is at least equal in washing and cleaning performance and cold water dispersability to the compositions described in the Kao specification which contain substantially higher levels of surfactant.

The aluminosilicate builder present in the compoistions of the invention may be crystalline or amorphous or a mixture thereof, and has the general formula

$0.8\text{-}1.5\ Na_20.Al_2O_3.0.8\text{-}6\ SiO_2$.

These materials contain some bound water and are required to have a calcium ion exchange capacity of at least about 50 mg CaO/g. The preferred aluminosilicates contain 1.5-3.5 SiO_2 units (in the formula above) and have a particle size of not more than about 100 microns, preferably not more than about 20 microns. Both amorphous and crystalline aluminosilicates can be made readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

Crystalline aluminosilicates (zeolites) are preferred for use in the present invention. Suitable materials are described, for example, in GB 1 473 201 (Henkel) and GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof. Especially preferred for use in the present invention is Type 4A zeolite.

The ratio of aluminosilicate builder (anhydrous basis) to total non-soap surfactant in the compositions of the invention is preferably within the range of from 1.2:1 to 1.8:1.

The non-soap surfactant present consists at least partially of anionic surfactant. Suitable anionic surfactants will be well known to those skilled in the art, and include linear alkylbenzene sulphonates, particularly sodium linear alkylbenzenesulphonates having an alkyl chain length of C_8-C_{15}; primary and secondary alkyl sulphates, particularly sodium C_{12}-C_{15} primary alcohol sulphates; alkyl ether sulphates; alpha-olefin and internal olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; fatty acid ester sulphonates; and combinations thereof.

If desired, the starting powder may contain nonionic surfactant, preferably in a minor amount. Nonionic surfactants too will be well known to those skilled in the art, and include primary and secondary alcohol ethoxylates, especially the C_{12}-C_{15} primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.

Suitably the surfactant component of the compositions of the invention may be constituted by 10 to 35 wt% of anionic surfactant and 0 to 10 wt% of nonionic surfactant.

Other types of non-soap surfactant, for example, cationic, zwitterionic, amphoteric or semipolar surfactants, may also be present if desired. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

If desired, soap may also be present, to provide foam control and additional detergency and builder power; soap is not included in the 17 to 35% figure for the total surfactant content of the compositions of the invention.

The compositions of the invention preferably do not contain more than 5 wt% of phosphate builders, and are more preferably substantially free of phosphate builders.

The particulate starting composition may be prepared by any suitable tower or non-tower method, for example, spray-drying or dry-mixing. If desired, the particulate starting material may be prepared at least partially by mixing in the high-speed mixer/granulator itself. The particulate starting material may consist at least partially of a spray-dried powder.

The final granulate has a bulk density of at least 650 g/litre and preferably at least 700 g/litre. It is also characterised by an especially low particle porosity, preferably not exceeding 0.25 and more preferably not exceeding 0.20, which distinguishes it from even the densest powders prepared by spray-drying alone.

The final granulate may be used as a complete detergent composition in its own right. Alternatively, it may be admixed with other components or mixtures prepared separately, and may form a major or minor part of a final product. Generally any additional ingredients such as enzymes, bleach and perfume that are not suitable for undergoing the granulation process may be admixed to the granulate to make a final product.

In one embodiment of the invention, for example, a detergent base powder is prepared by spray-drying an aqueous slurry of heat-insensitive and compatible ingredients; if desired, other ingredients may then be admixed; and the resulting powder is densified and granulated to give a product in accordance with the present invention. Yet further ingredients may if desired be admixed after granulation; the densified granulate of the invention may typically constitute from 40 to 100 wt% of a final product.

In another embodiment of the invention, a detergent base powder is prepared by dry mixing one or more raw materials and/or one or more premixes of raw materials, in the high-speed mixer/granulator itself or in other apparatus, and is then densified and granulated to give a product in accordance with the present invention. Again, further ingredients may if desired be added after granulation.

In yet another embodiment of the invention, the granulate prepared in accordance with the present invention is an "adjunct" comprising a relatively high level of detergent-active material on an inorganic carrier; and this may be admixed in a minor amount with other ingredients to form a final product.

The process

In the process of the invention, a particulate starting material (detergent base powder) prepared by any suitable method is granulated in a high-speed mixer/granulator to increase its bulk density and simultaneously to improve its powder properties. The process of the invention provides a route for the production of very dense granular detergent compositions, having excellent cleaning performance, containing low to moderate levels of anionic surfactant and high levels of aluminosilicate builder.

A preferred starting powder comprises:

(a) from 17 to 35 wt% of non-soap detergent-active material consisting at least partially of anionic detergent-active material, and

(b) from 28 to 45 wt% of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being from 0.9:1 to 2.6:1, and optionally other detergent components to 100 wt%.

On treatment in a high-speed mixer/granulator by the process of the invention, this gives a granular detergent composition or component in accordance with the first aspect of the invention. It will be noted, however, that the process of the invention also gives good results with compositions containing lower levels of detergent-active material.

In the process of the invention, granulation is effected by means of a high-speed mixer/granulator having both a stirring action and a cutting action. Preferably the stirrer and the cutter may be operated independently of one another, and at separately variable speeds. Such a mixer is capable of combining a high energy stirring input with a cutting action, but can also be used to provide other, gentler stirring regimes with or without the cutter in operation. It is thus a highly versatile and flexible piece of apparatus.

A preferred type of high-speed mixer/granulator for use in the process of the invention is bowl-shaped and preferably has a substantially vertical stirrer axis. Especially preferred are mixers of the Fukae (Trade Mark) FS-G series manufactured by Fukae Powtech Kogyo Co., Japan; this apparatus is essentially in the form of a bowl-shaped vessel accessible via a top port, provided near its base with a stirrer having a substantially vertical axis, and a cutter positioned on a side wall. The stirrer and cutter may be operated independently of one another, and at separately variable speeds.

Other similar mixers found to be suitable for use in the process of the invention are the Diosna (Trade Mark) V series ex Dierks & Söhne, Germany; and the Pharma Matrix (Trade Mark) ex T K Fielder Ltd., England. Other similar mixers believed to be suitable for use in the process of the invention include the Fuji (Trade Mark) VG-C series ex Fuji Sangyo Co., Japan; and the Roto (Trade Mark) ex Zanchetta & Co srl, Italy.

Another mixer found to be suitable for use in the process of the invention is the Lödige (Trade Mark) FM series batch mixer ex Morton Machine Co. Ltd., Scotland. This differs from the mixers mentioned above in that its stirrer has a horizontal axis.

As indicated above, the use of a high-speed mixer/granulator is essential in the process of the invention to effect granulation and densification. If desired, the mixer may also be used for a pretreatment step before granulation is carried out.

For example, it is within the scope of the invention, as previously indicated, for the particulate starting material to be prepared at least in part by mixing in the high-speed mixer/granulator. Thus, a dry-mixed starting powder may be prepared from its raw materials in the high-speed mixer/granulator; or one or more further ingredients may be admixed with an otherwise premixed powder prepared elsewhere (for example, by spray-drying). A suitable stirring/cutting regime and residence time may be chosen in accordance with the materials to be mixed.

Another possible pretreatment that may be carried out in the high-speed mixer/granulator is pulverisation; whether or not this is necessary depends, among other things, on the method of preparation of the starting powder and its free moisture content. Powders prepared by spray-drying, for example, are more likely to require pulverisation than powders prepared by dry-mixing. Again, the flexibility of the apparatus allows a suitable stirring/cutting regime to be chosen: generally relatively high speeds for both stirrer and cutter. A relatively short residence time (for example, 2-4 minutes for a 35 kg batch) is generally sufficient.

The essential feature of the process of the invention is the granulation step, during which densification to the very high values of at least 650 g/litre, preferably at least 700 g/litre occurs, giving a dense, granular product of very uniform particle size and generally spherical particle shape.

Granulation is effected by running the mixer at a relatively high speed using both stirrer and cutter; a relatively short residence time (for example, 5-8 minutes for a 35 kg batch) is generally sufficient. The final bulk density can be controlled by choice of residence time, and it has been found that the powder properties of the resulting granulate are not optimum unless the bulk density has been allowed to rise to at least 650 g/litre.

The presence of a liquid binder is necessary for successful granulation. The amount of binder added preferably does not exceed that needed to bring the free moisture content of the composition above about 6 wt%, since higher levels may lead to a deterioration in the flow properties of the final granulate. If necessary, binder, preferably water, may be added before or during granulation, but some starting powders will inherently contain sufficient moisture. If a liquid binder is to be added, it may be sprayed in while the mixer is running. In one preferred mode of operation, the mixer is first operated at a relatively slow speed while binder is added, before increasing the speed of the mixer to effect granulation.

If the starting powder has a sufficient free moisture content to render the addition of a binder unnecessary, pulverisation (if required) and granulation need not be regarded as separate process steps but as one single operation. Indeed, it is not, in that case, necessary to decide in advance whether or not pulverisation is required: the mixer may simply be allowed to do what is necessary, since the mixer conditions required are generally substantially the same for pulverisation and for granulation.

According to a preferred embodiment of the invention, granulation is carried out at a controlled temperature somewhat above ambient, preferably above 30° C. The optimum temperature is apparently formulation-dependent, but appears generally to lie within the range of from 30 to 45° C, preferably about 35° C.

It is an essential feature of the present invention that during granulation no "agent for improving surface properties" as defined in the above-mentioned JP 61 069897A (Kao) be present. When processing a formulation having a relatively high ratio of aluminosilicate builder to surfactant, in accordance with the present invention, the use of a finely divided particulate material such as fine sodium aluminosilicate during the granulation step is not only unnecessary but can with some formulations make granulation more difficult, or even impossible.

The optional flow aid

In accordance with a preferred embodiment of the invention, a finely divided particulate flow aid may be admixed with the granular material after granulation is complete. Advantageously, flow aid is added while the granulate is still in the high-speed mixer/granulator, and the mixer is operated at a slow speed for a further short period. No further granulation occurs at this stage. It is also within the scope of the invention to add the flow aid to the granulate after removing the latter to different apparatus.

This embodiment of the invention should be distinguished from the prior art process of JP 61 069897A (Kao), mentioned above, in which an "agent for improving surface properties", which can be fine sodium aluminosilicate, is present during the granulation stage itself. It is within the scope of the present invention to add a particulate flow aid after granulation is complete, but, as explained above, it is essential to the invention that no finely divided particulate "agent for improving surface properties" be present during granulation. The addition of a flow aid after granulation is complete can have an additional beneficial effect on the properties of the granulate, regardless of the formulation, whereas the presence of this type of material during the granulation step in the process of the invention makes processing more difficult.

The preferred granulation temperature of from 30 to 45°C, preferably about 35°C, may also be maintained during the subsequent admixture of a flow aid.

The flow aid is a finely divided particulate material. The preferred average particle size is 0.1 to 20 microns, more preferably 1 to 10 microns.

According to one preferred embodiment of the invention, the flow aid is finely divided amorphous sodium aluminosilicate, as described and claimed in our copending application of even date (Case C.3236). A suitable material is available commercially from Crosfield Chemicals Ltd, Warrington, Cheshire, England, under the trade mark Alusil. This material is effective in improving flow properties even at very low levels, and also has the effect of increasing bulk density. It is therefore possible to adjust bulk density by appropriate choice of the level of amorphous sodium aluminosilicate added after granulation.

Amorphous sodium aluminosilicate is advantageously used in an amount of from 0.2 to 5.0 wt%, based on the starting powder, more preferably from 0.5 to 3.0 wt%.

Another preferred flow aid is finely divided crystalline sodium aluminosilicate. The crystalline aluminosilicates discussed previously in the context of builders are also suitable for use as flow aids. They are, however, less weight-effective than the amorphous material and are suitably used in an amount of from 3.0 to 12.0 wt%, more preferably from 4.0 to 10.0 wt%.

If desired, both crystalline and amorphous sodium aluminosilicates may be used, together or sequentially, as flow aids.

Other flow aids suitable for use in the process of the invention include precipitated silica, for example, Neosyl (Trade Mark), and precipitated calcium silicate, for example, Microcal (Trade Mark), both commercially available from Crosfield Chemicals Ltd, Warrington, Cheshire, England.

The invention is further illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated.

EXAMPLES

Example 1

A detergent composition was prepared to the following composition by spray-drying an aqueous slurry to a free moisture content of substantially zero:

	parts
Linear alkylbenzene sulphonate	24.0
Nonionic surfactant	2.0
Soap	1.0
Zeolite (anhydr.)	38.0
Water bound with zeolite	10.84
Sodium silicate	4.0
Acrylate/maleate copolymer	2.0
Minor ingredients	2.0
Sodium carbonate	10.0
	94.64

It will be noted that the ratio of zeolite (anhydrous) to non-soap surfactant in this composition was 1.46.

35 kg of this spray-dried powder were introduced into a Fukae (Trade Mark) FS-G series high-speed mixer/granulator, and pulverised at high speed for 2-4 minutes. Water (2.0 parts) was then sprayed in while the mixer was allowed to run at a slower speed, then the speed was increased for 5-8 minutes while maintaining the temperature at about 35° C. During this period granulation occurred.

A sample of the granular product was removed from the mixer. It was free-flowing and showed no tendency to cake. Its dynamic flow rate was 65 ml/s.

1.0 part of Alusil (Trade Mark) fine amorphous sodium aluminosilicate was introduced into the Fukae mixer, which was then operated at a slow speed for 1 minute. The resulting granular product was free-flowing and showed no tendency to cake. Its bulk density was 740 g/litre and its particle porosity was less than 0.20. Its mean particle size was 405 microns and its dynamic flow rate was 105 ml/s.

The following ingredients were then mixed with the granular material to give 99 parts of final detergent powder:

Coloured speckles	1.5 parts
Enzyme (alcalase)	0.61 parts
Perfume	0.25 parts

Examples 2 and 3

20 kg of the spray-dried powder used in Example 1 were introduced into a Fukae (Trade Mark) FS-30 high-speed mixer/granulator, and pulverised for 4 minutes. Water (0.8 kg) was then added and the mixture granulated over a period of 4 minutes, while the temperature was maintained at about 35° C. A sample (Example 2) was removed from the mixer and its powder properties determined: these are shown in Table 1 below.

Alusil (Trade Mark) finely divided amorphous sodium aluminosilicate (0.2 kg) was then admixed. The physical properties of the resulting powder (Example 3) are shown in Table 1 below, from which the beneficial effect on flow and bulk density of adding a flow aid after granulation is complete is apparent. The presence of the Alusil did result in an increase in the content of fine particles < 180 microns, but not to an unacceptable level.

Comparative Example A

20 kg of the spray-dried powder used in Example 1 were introduced into the Fukae high-speed mixer/granulator, and pulverised for 4 minutes. Alusil (Trade Mark) finely divided amorphous sodium aluminosilicate (0.2 kg) was then introduced into the mixer. Water (0.8 kg) was then added and the mixture granulated over a period of 4 minutes, while the temperature was maintained at about 35° C. Physical properties of the resulting powder are shown in Table 1 below, from which the detrimental effect of adding Alusil before granulation are apparent. It will be noted that the increase in fines content is significantly greater when the Alusil is added before granulation.

Table 1

Example	2	3	A
Bulk density (g/l)	688	740	670
Dynamic flow rate (ml/s)	109	120	60
Particle size (microns)	550	480	380
Fines content (wt% of particles < 180 microns)	0	10	22
Particle porosity	0.1	0.1	not measured

Comparative Example B

This Example describes an attempt to carry out the process of the invention using a different spray-dried formulation containing a high level of anionic surfactant and a relatively low level of sodium aluminosilicate. The formulation was as follows:

	parts
Spary-dried base	
Linear alkylbenzene sulphonate	26.0
Primary alcohol sulphate	8.0
Nonionic surfactant	1.0
Soap	3.0
Zeolite (anhydrous)	14.8
Water bound with zeolite	4.2
Sodium silicate	6.0
Sodium carbonate (light soda ash)	5.0
Sodium sulphate	4.0
Acrylate/maleate copolymer	1.0
Free moisture	4.0
Postdosed	
Zeolite (hydrated)*	5.0
Sodium carbonate (granular)	15.0
Nonionic surfactant	3.0
	100.00

*equivalent to 3.9 parts of anhydrous zeolite

The ratio of zeolite (anhydrous) to non-soap surfactant (0.53) was less than 0.9:1.

The spray-dried base powder (7.7 kg) was placed in a Diosna (Trade Mark) bowl-type high-speed mixer/granulator, the 5.0 parts (0.3 kg) of nonionic surfactant were sprayed on, and the 15 parts (1.5 kg) of granular soda ash and the 5 parts (0.5 kg) of zeolite were added. Pulverisation at a stirrer speed of 196 rpm and a cutter speed of 3000 rpm was attempted for 1 minute, but the mixture overgranulated to form large lumps, and overheated.

Examples 4 and 5

35 kg of the spray-dried powder used in Example 1 were introduced into a Lödige (Trade Mark) FM series high-speed mixer/granulator, and pulverised for 4 minutes. Water (1.1 kg, 3.5%) was then sprayed in while the mixer continued to run at the same speed, then the mixer was allowed to run for a further 3 minutes while the temperature was maintained at about 35°C. During this period granulation occurred. A sample (Example 4) was removed from the mixer and its powder properties determined: these are shown in Table 2 below.

Alusil (Trade Mark) finely divided amorphous sodium aluminosilicate (1.2 kg) was then introduced into the mixer which was allowed to run for a further 0.5 minutes. Physical properties of the resulting powder (Example 3) are shown in Table 2 below; the results were similar to those obtained in Examples 2 and 3 using the Fukae mixer.

Comparative Example C

28.8 kg of the spray-dried powder used in Example 1 were introduced into a Lödige (Trade Mark) FM series high-speed mixer/granulator, and pulverised for 4 minutes. Alusil (Trade Mark) finely divided amorphous sodium aluminosilicate (1.2 kg) was then introduced into the mixer. Water (1.1 kg, 3.5%) was sprayed in while the mixer continued to run, then the mixer was allowed to run for a further 3 minutes while the temperature was maintained at about 35° C. During this period granulation occurred. Physical properties of the resulting powder are shown in Table 2 below.

Table 2

Example	4	5	C
Bulk density (g/l)	680	754	704
Dynamic flow rate (ml/s)	100	109	59
Particle size (microns)	573	524	424
Fines content (wt% of particles < 180 microns)	0	15	25
Particle porosity	<0.20	<0.20	not measured

Comparative Example D

This Example describes an attempt to carry out the process of the invention in the Lödige mixer using a spray-dried formulation, similar to that used in Comparative Example B, containing a high level of anionic surfactant and a relatively low level of sodium aluminosilicate. The formulation was as follows:

	parts
Linear alkylbenzene sulphonate	26.0
Primary alcohol sulphate	8.0
Nonionic surfactant	1.0
Soap	3.0
Zeolite (anhydr.)	10.9
Water bound with zeolite	3.1
Sodium silicate	6.0
Sodium carbonate	5.0
Sodium sulphate	4.0
Acrylate/maleate copolymer	1.0
Free moisture	2.9
	68.9

The ratio of zeolite to non-soap surfactant (0.31) was less than 0.9:1.

The formulation (30 kg) was placed in the Lödige FM mixer which was allowed to run for 4 minutes as in Examples 4, 5 and C. No dusty finely divided powder was obtained, and it was not clear that pulverisation was occurring. Water (1.05 kg, 3.5%) was then added, as in previous Examples, but attempted granulation for 0.5 minutes gave a dough.

Examples 6 to 8

Three powders were prepared by spray-drying the nominal composition shown in Example 1 to three different moisture contents, as shown in Table 3 below. Since the 38.0 parts of zeolite (anhydrous basis) in the formulation require 10.84 parts of water of hydration, the free moisture content of each powder is found by subtracting that figure from the total moisture content: it will be noted that the powder of Example 6 was overdried, while that of Example 8 contained 3.16 parts of free moisture.

A 10 kg batch of each powder was granulated (and, where necessary, pulverised) in a Diosna (Trade Mark) V series mixer, using a stirrer speed of 196 rpm and a cutter speed of 3000 rpm. In the case of Examples 6 and 7, where pulverisation initially occurred, water was sprayed into the mixer, in the amount and for the time indicated, before granulating. After granulation was complete, Alusil finely divided amorphous sodium aluminosilicate (0.1 kg) was admixed to the granulate. Properties of the three granulates are shown in Table 3.

Table 3

Example	6	7	8
Total moisture (wt %)	8.0	11.0	14.0
Pulverisation time (min)	4	3	-
Water addition:			
amount (kg)	0.2	0.1	-
time (min)	2	1	-
Granulation time (min)	4	3	4
Bulk density (g/l)	740	832	735
Dynamic flow rate (ml/s)	114	120	120
Compressibility (% v/v)	8.8	9.4	8.8
Particle size (microns)	483	553	521
Fines content (wt% < 180 microns)	5.0	2.4	6.1
Particle porosity	<0.20	<0.20	<0.20

Example 9

This Example describes a process in which an additional ingredient (sodium carbonate) was added to a spray-dried base powder in the mixer prior to pulverisation and granulation. The formulation was as follows:

Spray-dried base	parts
Linear alkylbenzene sulphonate	24.0
Nonionic surfactant	2.0
Soap	1.0
Zeolite (anhydr.)	38.0
Water bound with zeolite	10.84
Sodium silicate	4.0
Acrylate/maleate copolymer	2.0
Minor ingredients	2.0
	84.04
Added in mixer	
Sodium carbonate	10.0
	94.04

8.5 kg of the spray-dried base powder and 1.0 kg of sodium carbonate (light soda ash) were placed in the Diosna V100 mixer and were pulverised for 4 minutes at a stirrer speed of 196 rpm and a cutter speed of 3000 rpm. Water (0.45 kg) was added over a period of 2 minutes while the mixer was operated at a stirrer speed of 98 rpm and a cutter speed of 1500 rpm, then the mixture was granulated for 5 minutes at a stirrer speed of 196 rpm and a cutter speed of 3000 rpm. Finally Alusil (0.1 kg) was mixed in while the mixer was operated at a stirrer speed of 98 rpm with the cutter switched off.

The properties of the resulting granulate were as follows:

Bulk density (g/l)	780
Dynamic flow rate (ml/s)	133
Compressibility (%v/v)	7
Particle size (microns)	839
Particle porosity	0.10

The ratio of aluminosilicate to non-soap surfactant in the mixture subjected to granulation was 1.46.

Example 10

This Example describes a process in which the anionic surfactant was introduced in part via the spray-dried base powder and in part added to the base powder in the mixer prior to pulverisation and granulation. The formulation was as follows:

Spray-dried base	parts
Linear alkylbenzene sulphonate	12.0
Nonionic surfactant	2.0
Soap	1.0
Zeolite (anhydr.)	38.0
Water bound with zeolite	10.84
Sodium carbonate (light soda ash)	10.0
Sodium silicate	4.0
Acrylate/maleate copolymer	2.0
Minor ingredients	2.0
	81.84
Added in mixer	
Linear alkylbenzene sulphonate (powder)	12.0
	93.84

16.5 kg of the spray-dried base powder and 2.4 kg of linear alkylbenzene sulphonate powder (Marlon (Trade Mark) A390 ex Hüls) were placed in the Diosna V100 mixer and were pulverised for 4 minutes at a stirrer speed of 196 rpm and a cutter speed of 3000 rpm. Water (0.45 kg) was added over a period of 2 minutes while the mixer was operated at a stirrer speed of 98 rpm and a cutter speed of 1500 rpm, then the mixture was granulated for 5 minutes at a stirrer speed of 196 rpm and a cutter speed of 3000 rpm; during granulation the temperature rose to 40° C. Finally Alusil (0.19 kg) was mixed in while the mixer was operated at a stirrer speed of 98 rpm with the cutter switched off.

The properties of the resulting granulate were as follows:

Bulk density (g/l)	714
Dynamic flow rate (ml/s)	55
Compressibility (%v/v)	17
Particle size (microns)	712
Particle porosity	<0.20

The ratio of aluminosilicate to non-soap surfactant in the mixture subjected to granulation was 1.46.

Examples 11 and 12

These Examples involved a process in which a powder prepared by dry-mixing was densified and granulated in the high-speed mixer/granulator. The following formulation was prepared by mixing in a concrete mixer:

	parts
Linear alkylbenzene sulphonate	24.0
Nonionic surfactant	2.0
Soap	1.0
Zeolite (anhydr.)	38.0
Water bound with zeolite	10.84
Sodium carbonate (light soda ash)	10.0
Sodium silicate	4.0
Acrylate/maleate copolymer	2.0
Minor ingredients	2.0
	92.24

The ratio of aluminosilicate to non-soap surfactant in this mixture was 1.46.

20 kg of the formulation were placed in the Diosna V100 mixer aand mixed for 1 minute at a stirrer speed of 196 rpm and a cutter speed of 3000 rpm. Water (0.2 kg) was added over a period of 2 minutes while the mixer was operated at a stirrer speed of 98 rpm and a cutter speed of 1500 rpm, then the mixture was granulated for 4 minutes at a stirrer speed of 196 rpm and a cutter speed of 3000 rpm. A sample (Example 11) was removed and its powder properties measured (see below). Finally Alusil (0.2 kg) was mixed in while the mixer was operated at a stirrer speed of 98 rpm with the cutter switched off; and the powder properties of the final granulate (Example 12) were also measured.

The powder properties of the granulate before and after the addition of Alusil were as follows:

	11	12
Bulk density (g/l)	750	810
Dynamic flow rate (ml/s)	80	96
Compressibility (0/0v/v)	17.0	15.3
Particle size (microns)	-	607
Particle porosity	<0.20	<0.20

Example 13

The following formulation was prepared by a dry-mix process using the Fukae FS-1200 mixer:

	parts
Premix	
Linear alkylbenzene sulphonate*	25.0
Soap	1.0
Zeolite (hydrated)**	35.0
Sodium carbonate (light soda ash)	15.51
Sodium silicate	4.0
Acrylate/maleate copolymer	1.0
Minor ingredients	3.88

*Marlon A390 powder (90% active: figure given is corrected to 100% active).

**equivalent to 27.3 parts of anhydrous zeolite

Added in mixer	
Nonionic surfactant	2.0
Water	1.5
Postdosed	
Zeolite (hydrated)	10.0
	97.39

The ingredients listed under "Premix" were mixed for 2 minutes in the Fukae mixer at a stirrer speed of 80 rpm and a cutter speed of 2000 rpm. The nonionic surfactant was then added over a period of 1 minute, followed by water over a period of 2 minutes, while the mixer was operated at the same stirrer and cutter speeds. Granulation for 6 minutes at the same stirrer and cutter speeds followed, and finally the zeolite was added over a period of 2 minutes, still at the same stirrer and cutter speeds.

The properties of the resulting granulate were as follows:

Bulk density (g/l)	780
Dynamic flow rate (ml/s)	83
Compressibility (0/0v/v)	11.8
Particle size (microns)	477
Particle porosity	0.1

The ratio of zeolite (anhydrous) to non-soap detergent-active material in the material subjected to granulation was 1.0.

Claims

1. A granular detergent composition or component therefor, characterised in that it has a bulk density of at least 650 g/litre, and comprises:
(a) from 17 to 35 wt% of non-soap detergent-active material consisting at least partially of anionic detergent-active material;
(b) from 28 to 45 wt% (anhydrous basis) of crystalline or amorphous sodium aluminosilicate,
the weight ratio of (b) to (a) being from 0.9:1 to

2.6:1, and optionally other detergent components to 100 wt%.

2. A detergent composition or component as claimed in any preceding claim, characterised by a particle porosity of less than 0.25.

3. A detergent composition or component as claimed in claim 2, characterised by a particle porosity of less than 0.20.

4. A detergent composition or component as claimed in any preceding claim, characterised in that the ratio of (b) to (a) is within the range of from 1.2:1 to 1.8:1.

5. A detergent composition or component as claimed in any preceding claim, characterised in that the aluminosilicate (b) is a crystalline zeolite.

6. A detergent composition or component as claimed in any preceding claim, chracterised in that it contains form 0 to 5 wt% of phosphate builders.

7. A process for the preparation of a granular detergent composition or component having a bulk density of at least 650 g/litre, which is characterised by the step of treating a particulate starting material comprising:
(a) from 5 to 35 wt% of non-soap detergent-active material consisting at least partially of anionic detergent-active material, and
(b) from 28 to 45 wt% (anhydrous basis) of crystalline or amorphous sodium aluminosilicate, the weight ratio of (b) to (a) being at least 0.9:1, and optionally other detergent components to 100 wt%,
in a high-speed mixer/granulator having both a stirring action and a cutting action, in the presence of a liquid binder but in the absence of a finely divided particulate agent for improving surface properties, whereby granulation and densification to a bulk density of at least 650 g/litre are effected.

8. A process as claimed in claim 7, characterised in that granulation is carried out in a bowl-type high-speed mixer/granulator having a substantially vertical stirrer axis.

9. A process as claimed in claim 7 or claim 8, characterised in that the particulate starting material consists at least partially of spray-dried powder.

10. A process as claimed in any one of claims 7 to 9, characterised in that the particulate starting material is prepared at least in part by mixing in the high-speed mixer/granulator prior to granulation.

11. A process as claimed in any one of claims 7 to 10, characterised in that granulation effects an increase in bulk density to at least 700 g/litre.

12. A process as claimed in any one of claims 7 to 11, characterised by the further step of admixing a finely divided particulate flow aid to the granular material after granulation is complete.

13. A process as claimed in claim 12, characterised in that the flow aid is amorphous sodium aluminosilicate and is added in an amount of from 0.2 to 5.0 wt% based on the total composition.

14. A process as claimed in claim 12, characterised in that the flow aid is finely divided crystalline sodium aluminosilicate and is added in an amount of from 3.0 to 12.0 wt% based on the total composition.

15. A process as claimed in any one of claims 7 to 14, characterised in that the particulate starting material comprises:
(a) from 17 to 35 wt% of non-soap detergent-active material consisting at least partially of anionic detergent-active material, and
(b) from 28 to 45 wt% of crystalline or amorphous sodium aluminosilicate,
the weight ratio of (b) to (a) being from 0.9:1 to 2.6:1, and optionally other detergent components to 100 wt%.