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(54) Process for preparing detergent compositions.

(57) A process for the preparation of a high-bulk-density granular detergent composition or component comprises treating a particulate detergent composition in a high-speed mixer/granulator to effect granulation and densification to a bulk density of at least 650 g/litre, and subsequently admixing a small quantity of finely divided amorphous sodium aluminosilicate to the dense granular detergent composition thus obtained. This improves flow properties and raises bulk density further.

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Description

PROCESS FOR PREPARING DETERGENT COMPOSITIONS

TECHNICAL FIELD

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

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. For example, GB 1 517 713 (Unilever) 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.

EP 229 671A (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. In the Examples, the agent for improving surface properties is zeolite 4A (10 parts), used together with water (2 parts) as binder. After granulation, a further 3 parts of zeolite 4A are admixed with the product.

JP 84 041680B (Kao) describes a process in which a spray-dried detergent base powder is pulverised, mixed with powdery granules, having a crystallinity of 0-100% and a particle size of 0.1-300 microns, of sodium aluminosilicate, calcium silicate, calcium carbonate, magnesium silicate or sodium carbonate, and simultaneously or subsequently mixed with a tackifying substance, for example, a nonionic surfactant, alkyl ether sulphate or higher alcohol.

EP 220 024A (Procter & Gamble) discloses a process for 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 crystalline sodium aluminosilicate and sodium carbonate) being added before compaction and/or after granulation.

The present inventors have now discovered that detergent compositions of high bulk density and excellent flow properties can be prepared by granulating a spray-dried or dry-mixed detergent base powder in a high-speed mixer/granulator and then admixing a small amount of finely divided amorphous aluminosilicate after granulation is complete. The amorphous material is substantially more weight-effective than crystalline zeolite for this purpose.

DEFINITION OF THE INVENTION

The present invention accordingly 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 steps of:

(i) treating a particulate material comprising one or more non-soap detergent-active compounds and one or more inorganic builders in a high-speed mixer/granulator having both a stirring action and a cutting action, in the presence of a liquid binder, whereby granulation and densification to a bulk density of at least 650 g/litre are effected,

(ii) admixing finely divided amorphous sodium aluminosilicate to the granular material obtained in step (i).

DETAILED DESCRIPTION OF THE INVENTION

The process

In the process of the invention, a particulate starting material (detergent base powder) prepared by any suitable method is treated 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 and good powder properties.

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 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 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.

An 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.

In accordance with the invention, finely divided amorphous sodium aluminosilicate is admixed with the granular material after granulation is complete. Advantageously, the amorphous sodium aluminosilicate 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 amorphous sodium aluminosilicate to the granulate after removing the latter to different apparatus.

The granulation stage is preferably 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. This temperature may also be maintained during the admixture of the finely divided amorphous sodium aluminosilicate.

The amorphous sodium aluminosilicate

The amorphous sodium aluminosilicate used in the process of the present invention is a finely divided particulate material. The preferred average particle size is 0.1 to 20 microns, more preferably 1 to 10 microns. A suitable material is available commercially from Crosfield Chemicals Ltd, Warrington, Cheshire, under the trade mark Alusil.

The 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%.

This material is effective even at very low levels in improving flow properties, 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 aluminosilicate added after granulation.

The amorphous material used in the process of the invention should be distinguisbed from zeolite (hydrated crystalline sodium aluminosilicate) which is substantially less weight-effective in the context of the present invention. Substantially higher levels than those quoted above are needed before any comparable flow or bulk density benefit is observed.

The starting powder

The process of the invention may be used to densify and improve any detergent powder prepared by any 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 process of the invention has been found to give especially satisfactory results with detergent base powders containing low to moderate levels of surfactant and relatively high levels of inorganic builder.

According to a first preferred embodiment of the invention, the process is used for the preparation of high-bulk-density powders containing substantial levels of sodium aluminosilicate builder. These powders preferably contain not more than 5 wt% of phosphate builders, and are more preferably substantially free of phosphate builders.

Thus a preferred starting powder might comprise:

(a) from 5 to 35 wt% of non-soap 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%.

The process of the invention is outstandingly suitable for preparing the high-bulk-density powders, containing moderate levels of surfactant and high levels of zeolite, described and claimed in our copending application of even date (Case C.3235). These powders comprise:

(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% (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%.

The aluminosilicate builder present in the starting powder may be crystalline or amorphous or a mixture thereof, and has the general formula

$0.8\text{-}1.5\ Na_2O.Al_2O_3.0.8\text{-}6\ SiO_2$.

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 in the low- or zero-phosphate starting powders treated by the process of 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. Type 4A zeolite is especially preferred.

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

According to a second preferred embodiment of the invention, the process is used for the preparation of high-bulk-density powders containing substantial levels of water-soluble inorganic salts, including sodium tripolyphosphate and/or sodium carbonate, as described and claimed in our copending application of even date (Case C.3261).

Thus a preferred starting powder might comprise:

(x) from 12 to 70 wt% of non-soap detergent-active material, and

(y) at least 15 wt% of water-soluble inorganic salts, including sodium tripolyphosphate and/or sodium carbonate, the weight ratio of (y) to (x) being at least 0.4:1, and optionally other detergent components to 100 wt%.

Preferably the weight ratio of (y) to (x) is within the range of from 0.4:1 to 9:1, more preferably from 1:1 to 9:1. An especially preferred starting powder contains from 15 to 70 wt% of water-soluble inorganic salts, more preferably from 15 to 50 wt%, especially from 20 to 40 wt%, of sodium tripolyphosphate.

In both the first and second preferred embodiments of the invention, the non-soap surfactant present in the starting powder preferably 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. 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 starting powder may be constituted by from 0 to 70%, preferably from 8 to 60% by weight, of anionic surfactant, and from 0 to 20%, preferably from 0 to 10%, by weight 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 figures for the surfactant content quoted previously.

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, not exceeding 0.25 and 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.

A detergent base powder may, for example, be 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 densified and granulated in accordance with the present invention. Yet further ingredients may if desired be admixed after granulation; the densified granulate may typically constitute from 40 to 100 wt% of a final product.

Alternatively, a detergent base powder may be 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 then densified and granulated in accordance with the present invention. Again, further ingredients may if desired be added after granulation.

Yet again, the granulate prepared in accordance with the present invention may be 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 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 having a bulk density of 350 g/litre was prepared to the following composition by spray-drying an aqueous slurry:

	%
Linear alkylbenzene sulphonate	20.0
Nonionic surfactant	2.0
Soap	1.0
Zeolite (anhydr.)	35.0
Water with zeolite	10.0
Sodium silicate	4.0
Sodium succinate	2.0
Acrylate/maleate copolymer	2.0
Sodium sulphate	10.45
Sodium carbonate	10.0
Minor ingredients	1.55
Free moisture	2.0
	100.0

It will be noted that the ratio of zeolite (anhydrous) to non-soap surfactant in this composition was 1.59.

25 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 (stirrer 180 rpm, cutter 3013 rpm) for 4 minutes at 32°C. Water (500 g, 2.0%) was then sprayed in over a period of 0.5 min while the mixer was allowed to run at a slower speed (stirrer 100 rpm, cutter 3000 rpm) at a temperature of 35°C. The mixer was then allowed to run at an stirrer speed of 140 rpm, a cutter speed of 2700 rpm and a temperature of 36-37°C to effect granulation.

After granulation was complete, Alusil (Trade Mark) fine amorphous sodium aluminosilicate (250 g, 1%) was introduced into the Fukae mixer, which was then operated at a slow speed (stirrer 90 rpm, cutter 300 rpm) for 1 minute. The resulting granular product was free-flowing and showed no tendency to cake. Its properties, and those of a sample removed before the addition of the Alusil, are shown in Table 1 below.

It will be noted that the final mean particle size after Alusil addition was slightly smaller than before the addition of the Alusil, showing that a minor amount of degranulation had occurred during this treatment.

Surprisingly, however, the percentage of fine particles had decreased. The substantial increase in bulk density effected by the addition of Alusil will be noted.

Table 1

	Before Alusil addition	After Alusil addition
Bulk density (g/l)	794	892
Mean particle size (microns)	785	704
Dynamic flow rate (ml/s)	25	120
Fines (wt% of particles <180 microns)	13	8
Particle porosity	<0.20	<0.20

Examples 2 & 3, Comparative Examples A & B

In these Examples, the effect of adding Alusil after granulation was compared with the effect of adding crystalline zeolite 4A after granulation. Samples of the spray-dried powder used in Example 1 were treated in the Fukae mixer as described in that Example, Alusil or zeolite as shown in Table 2 below being added after granulation was complete.

Table 2

Example	2	3	A	B
Flow aid (wt %):				
Alusil	1.0	1.7	-	-
Zeolite	-	-	1.0	3.0
Bulk density (g/l)	856	854	740	784
Dynamic flow rate (ml/s)	92.3	92.3	33.5	42.5
Particle size (microns)	507	546	530	529
Fines content (wt% <180 microns)	11	8	2	1

The large differences in both bulk density and dynamic flow rate will be noted.

Example 4

This Example describes the preparation of a complete detergent product using the process of the invention.

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 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. The mixer was then stopped, water (2.0 parts) was sprayed in, and the mixer was then restarted at a slower speed and allowed to run for 5-8 minutes while the temperature was maintained at about 35°C; during this period granulation occurred.

A sample of the granular product was removed from the Fukae 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 100 parts of final detergent powder:

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

Example 5, Comparative Example C

35 kg of the spray-dried powder used in Example 4 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 (Comparative Example C) was removed from the mixer and its properties are shown in Table 3 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. The properties of the resulting powder (Example 5) are shown in Table 3 below, from which the benefits of adding a flow aid after granulation is complete are 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 D

28.8 kg of the spray-dried powder used in Example 4 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. The properties of the resulting powder are shown in Table 3 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 3

Example	C	5	D
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 E

The procedure of Comparative Example D was repeated, but the Alusil was added before the pulverisation step instead of after it. Pulverisation and granulation were carried out as in previous Examples, but the resulting product had a dynamic flow rate of zero.

Example 6, Comparative Example F

20 kg of the spray-dried powder used in Example 4 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 (Comparative Example F) was removed from the mixer and its powder properties determined: these are shown in Table 3 below.

Alusil (Trade Mark) finely divided amorphous sodium aluminosilicate (0.2 kg) was then admixed. The physical properties of the resulting powder (Example 6) are shown in Table 3 below; the results were similar to those obtianed in Examples 4 and C using the Lödige mixer.

Comparative Example G

20 kg of the spray-dried powder used in Example 4 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 3 below: the results were similar to those obtained using the Lödige mixer (Comparative Example D).

Table 3

Example	6	F	G
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

Example 7, Comparative Example H

These Examples involved a process in which a powder prepared by dry-mixing was densified and granulated in a 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 this formulation were placed in a Diosna (Trade Mark) 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 (Comparative Example H) 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 7) were also measured.

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

	H	7
Bulk density (g/l)	750	810
Dynamic flow rate (ml/s)	80	96
Compressibility (%v/v)	17.0	15.3
Particle size (microns)	-	607
Particle porosity	<0.20	<0.20

Example 8, Comparative Example J

A detergent powder built with sodium tripolyphosphate was prepared by spray-drying an aqueous slurry to the following formulation:

	wt %
Linear alkylbenzene sulphonate	9.7
Nonionic surfactant	2.8
Soap	4.9
Sodium tripolyphosphate	42.5
Sodium sulphate	14.8
Sodium silicate	10.0
Minor ingredients	2.8
Water	12.5
	100.0

The ratio of water-soluble crystalline inorganic salts (sodium tripolyphosphate and sodium sulphate) to non-soap surfactant was 4.6:1.

Two separate 20 kg batches of this powder were densified in the Fukae mixer, as follows. The powder was initially subjected to a 2-3 minute warming-up period, with the stirrer running at 50 rpm and with the cutter switched off, until the temperature had reached about 30-35°C. Pulverisation for 0.5 minutes at a stirrer speed of 180 rpm and a cutter speed of 1000 rpm followed; water (0.5 wt%) was added over 0.5 minutes while the mixer was operated at a stirrer speed of 100 rpm and a cutter speed of 3000 rpm; then granulation was carried out for 6 minutes at a stirrer speed of 140 rpm and a cutter speed of 3000 rpm.

To the first sample (Example 8), Alusil (1.5 wt%) was added over a period of 1 minute while the mixer was operated at a stirrer speed of 90 rpm and a cutter speed of 300 rpm.

To the second sample (Comparative Example J), zeolite (5 wt%) was added over the same period of time and under the same mixer conditions.

Powder properties were as follows:

	8	J
Yield <1700 microns (wt%)	93	97
Average particle size (microns)	555	480
Bulk density (g/litre)	840	780
Dynamic flow rate (ml/s)	92	61
Compressibility (%v/v)	7	12
Particle porosity	<0.20	<0.20

Example 9, Comparative Example K

This Example illustrates the sequential addition of zeolite and Alusil to a densified powder.

A detergent base powder 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 4A (anhydrous))	(27.27
Water with zeolite)	(7.73
Sodium silicate	4.0
Acrylate/maleate copolymer	1.0
Minor ingredients	2.85
Sodium carbonate	16.51

The weight ratio of anhydrous zeolite to non-soap detergent in this base powder was 1.05:1.

90 parts of this base powder were granulated and densified in the Fukae mixer as described in previous Examples, then a further 10 parts (hydrated basis) of zeolite 4A were admixed. A sample (Comparative Example K) was removed, and then Alusil (0.4 parts) was added to give a final granulate (Example 9).

The powder properties are shown below, and demonstrate that the final addition of Alusil, in the small quantity of 0.5 parts to 100, effected significant increases in bulk density and flow rate, and decreases in cohesiveness and caking, even though 10 parts of zeolite had already been postdosed.

	K	9
Bulk density, g/l	732	760
Dynamic flow rate, ml/s	85	98
Powder cohesiveness (%)	10	9
Powder caking after storage in cartons for 6 months (%)	10	5
Particle porosity	<0.20	<0.20

Claims

1. A process for the preparation of a granular detergent composition or component having a bulk density of at least 650 g/litre, characterised by the steps of:
   (i) treating a particulate material comprising one or more non-soap detergent-active compounds and one or more inorganic builders in a high-speed mixer/granulator having both a stirring action and a cutting action, in the presence of a liquid binder, whereby granulation and densification to a bulk density of at least 650 g/litre are effected,
   (ii) admixing finely divided amorphous sodium aluminosilicate to the granular material obtained in step (i).
2. A process as claimed in claim 1, characterised in that the finely divided amorphous sodium

aluminosilicate is added in an amount of from 0.2 to 5.0 wt%, based on the total composition.

3. A process as claimed in any preceding claim, characterised in that step (ii) is also carried out in the high-speed mixer/granulator.

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

5. A process as claimed in any preceding claim, characterised in that the particulate starting material consists at least partially of spray-dried powder.

6. A process as claimed in any preceding claim, characterised in that the particulate starting material is prepared at least in part by mixing in the high-speed mixer/granulator prior to granulation.

7. A process as claimed in any preceding claim, characterised in that the particulate starting material comprises:(a) from 5 to 35 wt% of non-soap 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%.

8. A process as claimed in claim 7, 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% (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%.

9. A process as claimed in claim 7 or claim 8, characterised in that the aluminosilicate (b) in the particulate starting material is a crystalline zeolite.

10. A process as claimed in any one of claims 1 to 6, characterised in that the particulate starting material comprises:
(x) from 12 to 70 wt% of non-soap detergent-active material, and
(y) at least 15 wt% of water-soluble crystalline inorganic salts, including sodium tripolyphosphate and/or sodium carbonate,
the weight ratio of (y) to (x) being at least 0.4:1, and optionally other detergent components to 100% by weight.

11. A process as claimed in claim 10, characterised in that the particulate starting material has a ratio of (y) to (x) within the range of from 1:1 to 9:1.

12. A process as claimed in claim 10 or claim 11, characterised in that the particulate starting material comprises from 15 to 70 wt% of crystalline inorganic salts including sodium tripolyphosphate and/or sodium carbonate.

13. A process as claimed in claim 12, characterised in that the particulate starting material comprises from 15 to 50 wt% of sodium tripolyphosphate.

European Patent
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EUROPEAN SEARCH REPORT

Application Number

EP 89 30 4205

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.4)
Y	DE-A-3 617 756 (LION CORP.) * whole document *	1,7-13	C 11 D 11/00 C 11 D 3/08 C 11 D 17/06
A	---	2	
Y	DATABASE WPIL no. 133904, 1986, Derwent Publication; & JP - A - 61 069897 (KAO CORP.) 10.04.1988 * abstract *	1,7-13	
A	idem ---	3-6	
A,P	DATABASE WPIL no. 6662309, 1989, Derwent Publication; & JP - A - 1020298 (KAO CORP.) 24.01.1989 * abstract * ---	1-13	
A	PATENT ABSTRACTS OF JAPAN vol. 2, no. 59, 27 April 1978; & JP - A - 53 16713 (LION YUSHI K.K.) 16.02.1978 * abstract * ---	1,2,10-13	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
A	FR-A-1 594 106 (GROSSEINKAUFS-GESELLSCHAFT DEUTSCHER KONSUMGENOSSENSCHAFTEN MBH) * claims * -----	1-13	C 11 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	19-07-1989	PELLI-WABLAT B

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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