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(54) **Detergent powders and process for preparing them.**

(57) A spray-dried zero-phosphate powder suitable as a base for a detergent composition contains anionic surfactant, optional nonionic surfactant, aluminosilicate builder, polymeric polycarboxylate and a low or zero level of electrolyte, preferably sodium carbonate. The powder has a particle porosity of less than 0.40. 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 detergent compositions of very high bulk density.

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DETERGENT POWDERS AND PROCESSES FOR PREPARING THEM

TECHNICAL FIELD

The present invention relates to spray-dried zero-phosphate powders suitable for use as detergent compositions or components thereof. The powders of the invention are built with crystalline or amorphous sodium aluminosilicate.

BACKGROUND AND PRIOR ART

Following the move in many countries of the world to reduce or eliminate phosphate builders in detergents, sodium aluminosilicates, both crystalline (zeolite) and amorphous (NAS), have become well known as detergency builders. These materials do not, however, possess an ability comparable to that of sodium tripolyphosphate to contribute to the structure of a spray-dried powder. Alkali metal silicates are frequently included in detergent powders as structurants, to reduce washing machine corrosion and to increase alkalinity. It is well known, however, that if aluminosilicate and silicate are together in a detergent slurry they can interact unfavourably: agglomeration occurs to give powders containing large particles which are slow to disperse in the wash liquor, giving reduced washing performance. Thus the inferior structuring ability of aluminosilicates cannot simply be compensated for by including larger quantities of sodium silicate in the slurry.

Recently detergent manufacturers have been attempting to prepare detergent powders of increased bulk density, for example, 600 g/litre and above as opposed to the 400-500 g/litre of current conventional powders. This trend imposes additional constraints and requirements on the detergents formulator.

We have now discovered that spray-dried zeolite-built powders of very high bulk density may be prepared by spray-drying slurries of defined moisture content, and low or zero levels of electrolyte. The presence of a polymeric polycarboxylate is also essential to provide building and structuring. The powders are characterised by exceptionally low particle porosity and excellent powder properties. If desired, the bulk density can be increased further, and powder properties enhanced, by postdosing high-bulk-density salts, notably sodium sulphate, to the spray-dried powder.

Spray-dried powders containing anionic surfactants, built with zeolite and polymeric polycarboxylate and containing inorganic salts such as sodium carbonate and sodium sulphate, are disclosed in EP 137 669A (Procter & Gamble), EP 209 840A (Henkel), EP 63 399B (Procter & Gamble) and GB 2 095 274 (Colgate-Palmolive), but these powders do not have the low electrolyte levels characteristic of the present invention.

DEFINITION OF THE INVENTION

The present invention provides a spray-dried zero-phosphate detergent powder comprising:

(a) from 5 to 60 by weight, in total, of one or more anionic detergent-active compounds;
(b) optionally from 0 to 30% by weight of one or more nonionic detergent-active compounds:
(c) from 15 to 86% by weight of crystalline or amorphous sodium aluminosilicate builder;
(d) from 2 to 40% by weight of a polymeric polycarboxylate;
(e) optionally sodium carbonate provided that if the amount of anionic detergent-active compound (a) exceeds 14.5% by weight the weight ratio of sodium carbonate (e) to anionic detergent-active compound (a) does not exceed 1.1:1;
(f) optionally other salts;
(g) optionally conventional minor ingredients; the composition having a total electrolyte level not exceeding 25% by weight and a particle porosity of less than 40%.

The present invention further provides a process for the preparation of a zero-phosphate detergent powder as defined above, which comprises spray-drying an aqueous slurry to form a powder having a particle porosity of 40% or less, the slurry comprising:

(a) one or more anionic detergent-active compounds, in an amount of from 5 to 60% by weight based on the powder,

(b) optionally one or more nonionic detergent-active compounds, in an amount of from 0 to 30% by weight based on the powder,

(c) a crystalline or amorphous sodium aluminosilicate builder, in an amount of from 15 to 86% by weight based on the final powder,

(d) a polymeric polycarboxylate, in an amount of from 2 to 40% by weight based on the final powder,

(e) optionally sodium carbonate provided that if the amount of anionic detergent-active compound (a) exceeds 14.5% by weight the weight ratio of sodium carbonate (e) to anionic detergent-active compound (a) does not exceed 1.1:1;

(f) optionally other salts provided that the total electrolyte level of the powder does not exceed 25% by weight,

(g) optionally conventional minor ingredients.

DESCRIPTION OF THE INVENTION

The present invention is concerned with a spray-dried detergent powder containing anionic surfactant which is suitable for use as a base for a detergent composition, or indeed as a whole detergent composition in its own right. For convenience this material will be referred to hereinafter as the base powder of the invention. It is characterised by an exceptionally low particle porosity of less than 40%, preferably less than 35%. The particle porosity may be determined by the recognised technique of mercury porosimetry.

It is well known that the presence of anionic surfactant in a slurry tends to cause entrainment of air or "puffing" during spray-drying so that it is very difficult to obtain powders having low particle porosity. It is much easier to prepare to low porosities powders containing no surfactant, or only nonionic surfactant. We have now found that low-porosity powders containing appreciable amounts of anionic surfactant can be prepared by spray-drying, provided that the amount of electrolyte (salts) is kept below a certain level.

The total level of electrolyte (salts) in the base powder of the invention does not exceed 25% by weight. Preferably the electrolyte level does not exceed 20%, more preferably 15% and most preferably 13%. Advantageously the electrolyte present is constituted substantially wholly by sodium carbonate. The lowest particle porosities - around 25% - are obtained when no electrolyte whatsoever is present. Surprisingly, however, it has been found that when a fluorescer is present, the presence of a low level of sodium carbonate is beneficial in reducing or eliminating discolouration (yellowing), while giving particle porosities that are not substantially greater than those obtained at zero electrolyte. The preferred amount of sodium carbonate present for this benefit is from 2 to 20% by weight, preferably from 5 to 15% by weight, of the spray-dried powder.

The presence of silicate in the slurry has been found not to be essential: surprisingly the spray-dried particles obtained are strong enough to give excellent powder properties. Any silicate required in order to prevent washing machine corrosion can therefore be postdosed, for example as described in EP 240 356A (Unilever), to be published on 7 October 1987, and EP 242141A (Unilever), to be published on 21 October 1987, thus avoiding the problem of unfavourable interaction with aluminosilicate in the slurry. If desired, however, the slurry may contain sodium silicate, but preferably in an amount not exceeding 10% by weight, and more preferably not exceeding 5% by weight, based on the spray-dried powder.

Salts other than sodium carbonate may if desired be present. It is preferred, however, that the slurry should be virtually free of sodium sulphate, other than the small quantities inevitably associated as impurities with other components such as anionic surfactants. Sodium sulphate levels as low as 25% (based on the powder) give particles of considerably greater porosity. Similar considerations apply to other inorganic salts: it is therefore preferred that no significant amounts of salts other than sodium carbonate and possibly sodium silicate be present.

The powder of the invention contains crystalline or amorphous aluminosilicate, preferably zeolite, in an amount of from 15 to 86% by weight. The preferred level is from 20 to 70% by weight, more preferably from 30 to 70% by weight. This component is the principal builder in the powder of the invention.

As auxiliary builder there is also present a polymeric polycarboxylate in an amount from 2 to 40% by weight, preferably from 3 to 25% by weight, and more preferably from 4 to 15% by weight. The polymer appears to be essential not only for building but also for creating adequately structured spray-dried particles. Suitable polymer include the following, the list not being exhaustive:

salts of polyacrylic acid, for example, Versicol (Trade Mark) E5, E7 and E9 ex Allied Colloids, average molecular weights 3500, 27 000 and 70 000 respectively; Narlex (Trade Mark) LD 30 and 34 ex National Adhesives and Resins Ltd, average molecular weights 5000 and 25 000 respectively; Acrysol (Trade

Mark) LMW-10, LMW-20, LMW-45 and A-IN ex Rohm & Haas, average molecular weights 1000, 2000, 4500 and 60 000; and Sokalan (Trade Mark) PA 110S ex BASF, average molecular weight 250 000;

ethylene/maleic acid copolymers, for example, the EMA (Trade Mark) series ex Monsanto;

methyl vinyl ether/maleic acid copolymers, for example Gantrez (Trade Mark) AN119 ex GAF Corporation;

acrylic acid/maleic acid copolymers, for example, Sokalan (Trade Mark) CP5 and CP7 ex BASF, average molecular weights 70 000 and 50 000 respectively; and

acrylic phosphinates, for example, the DKW range ex National Adhesives and Resins Ltd or Belsperse (Trade Mark) 161 ex Ciba-Geigy AG, as disclosed in EP 182 411A (Unilever).

Mixtures of two or more of these polymers may of course be used if desired.

The spray-dried base powders of the invention also contain as essential ingredients one or more anionic detergent-active compounds (surfactants), in an amount of from 5 to 60% by weight, preferably from 10 to 60% by weight, more preferably from 10 to 30% by weight, and especially from 12 to 30% by weight. At higher anionic surfactant levels, of 14.5% by weight and above, we have found that it is necessary that the weight ratio of sodium carbonate to anionic surfactant should not exceed 1.1:1 in order for particle porosities of 40% and below to be obtained. At lower anionic surfactant levels, however, higher ratios can be tolerated: only the limitation on the total electrolyte level (maximum 25% by weight) need be considered.

Anionic surfactants are well known to those skilled in the detergents art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an average chain length of about C_{12} ; primary and secondary alcohol sulphates, particularly sodium C_{12} -C_{15} primary alcohol sulphates; olefin sulphonates; alkane sulphonates; and fatty acid ester sulphonates.

If desired, one or more nonionic detergent-active agents (surfactants) may also be present in the spray-dried base powders of the invention, in an amount not exceeding 30% by weight, and preferably not exceeding 15% by weight. A preferred range for nonionic surfactant content is from 1 to 10% by weight. The weight ratio of anionic surfactant to nonionic surfactant is preferably at least 0.67:1, more preferably at least 1:1.

Nonionic surfactants that may be used include the 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.

The spray-dried powder of the invention may if desired contain one or more soaps of fatty acids, but the level of soap present is preferably not high enough to cause a significant reduction in particle porosity.

The total amount of detergent-active material, including soap, in the spray-dried base powders of the invention is preferably within the range of from 10 to 75% by weight, more preferably from 12 to 65% by weight. For powders intended for use in European front-loading automatic washing machines the especially preferred range is from 12 to 50% by weight, with a weight ratio of anionic surfactant to nonionic surfactant within the range of from 1:1 to 10:1.

The spray-dried base powder may of course contain any of the usual minor ingredients suitable for undergoing slurry-making and spray-drying processes, for example, antiredeposition agents and fluorescers.

The spray-dried base powder will also contain a certain amount of moisture: both moisture bound to the zeolite (about 1 part of bound water to 4 parts of zeolite) and free moisture. The free moisture content influences powder porosity and can be determined by suitable choice of spray-drying conditions. The higher the free moisture content, the lower the porosity will be, but powder properties (flow, resistance to caking, compressibility, agglomerate strength) deteriorate as the moisture content rises. A convenient expression of powder moisture content is the relative humidity. This is the partial water vapour pressure of a powder sample in a relatively small closed container at 20°C, expressed as a percentage of the partial water vapour pressure of the atmosphere at that temperature. Spray-dried base powders of the present invention preferably have relative humidities not exceeding 70%, more preferably within the range of from 45 to 65%.

The base powder of the invention is prepared, as indicated above, by spray-drying an aqueous slurry of the ingredients. The term "spray-drying" here is not restricted to high-temperature operation but also includes processes, more commonly referred to as spray-cooling, in which a tower inlet temperature lower than about 100-150°C is used.

If desired, the drying process may be carried out in two stages. For example, the slurry may be spray-dried to a relatively high powder moisture content, and the resulting wet powder dried further to its final desired moisture content using other apparatus, for example, a fluidised bed as described in GB 1 237 084 (Unilever).

As well as having low particle porosity, the base powders of the invention are characterised by excellent agglomerate strength. This is defined as the pressure that has to be exerted on a sample of powder to compress it to a bed porosity of 0.4. The latter value has been selected since it is known to be the bed porosity of densely packed granular solids, including detergent powders: to achieve bed porosities below 0.4 any agglomerates in a powder sample have to be broken down into the primary particles of which they are composed, these primary particles corresponding in size to the droplets formed when the detergent slurry was atomised in the spray-drying tower. Thus the agglomerate strength, as its name implies, is a measure of the resistance of the agglomerates in a powder, on compression, to breakdown into the smaller primary particles.

The agglomerate strength is measured as follows. A 0.3 g sample of the 250-500 μm sieve fraction of the spray-dried base powder is subjected to compression in a compression cell of circular cross-section, having a diameter of 1.3 cm and (hence) a cross-sectional area of 1.33 cm^2. The work of compression is measured and plotted against the height of the powder bed in the cell.

Bed porosity as a function of bed height can be calculated from the bulk density of the powder bed (calculated from the weight of the powder sample, its height and the cross-sectional area of the compression cell), the true density of the material (solid density) and the powder porosity, by means of the following equation:

$$\text{Bulk density} = \text{solids density} \times \left[1 - \text{particle porosity}\right]\left[1 - \text{bed porosity}\right]$$

The particle porosity, as previously mentioned, may be determined by mercury porosimetry.

These relationships enable a bed height corresponding to a bed porosity of 0.4 to be determined, and hence the compression required to achieve that bed height: that is the agglomerate strength.

Agglomerate strength values depend on bulk density as well as on the formulation of the slurry, and for powders having bulk densities of 400 g litre or above values above 7 N/cm^2 are considered good.

The spray-dried base powder of the invention is useful as a detergent composition in its own right. Generally, however, various additional ingredients may be sprayed on or postdosed to give a more efficient product, and the base powder may form a major or minor part of a more complex composition. Thus granular detergent compositions in accordance with the present invention may contain, for example, from 10 to 100% of the spray-dried base powder of the invention. Typically, such detergent compositions may contain from 10 to 90% by weight, preferably from 30 to 90% by weight, of base powder and from 10 to 90% by weight, preferably from 10 to 70% by weight, of postdosed solid and/or liquid material. The term "postdosing" here encompasses any non-spray-drying method by which solid or liquid ingredients may be added to the base powder, for example, dry mixing, granulation, agglomeration, overspraying or any combination of these techniques.

Some materials may be postdosed because they are sensitive to heat and thus unsuitable for undergoing spray-drying. Examples of such materials include surfactants, enzymes, bleaches, bleach precursors, bleach stabilisers, lather suppressors, perfumes and dyes. Liquid or pasty ingredients may conveniently be adsorbed on to a solid, porous, particulate, generally inorganic, carrier which is then postdosed to the base powder of the invention. Examples of such ingredients are anionic and nonionic surfactants, and liquid lather suppressors such as silicone oil.

Another material that may be postdosed, as previously mentioned, is solid sodium silicate: thus unfavourable interaction with the sodium aluminosilicate in the slurry is avoided. Alternatively an aqueous solution or dispersion of sodium silicate may be blown into the tower simultaneously with the introduction of the base powder slurry, as described and claimed in our European Patent Application No. 87 308239.0 filed on 17 September 1987.

As mentioned previously, the base powder of the invention is characterised by an especially low particle porosity of less than 40%, preferably less than 35%. Its bulk density is therefore very high and it is exceptionally suitable for use as a base for granular detergent compositions of high bulk density, for example, of 650 g/litre and above, especially from 700 to 900 g/litre. In such compositions, it is important that any postdosed material does not lower the bulk density significantly.

One especially preferred embodiment of the invention is a very high bulk density detergent composition obtained by filling the voids between the relatively large particles of base powder with postdosed solid material, preferably including a substantial proportion of sodium sulphate, in the form of a finely divided dense powder of low porosity. In this manner an increase in bulk density of 150 g/litre or more, preferably at least 200 g/litre, may be achieved.

In this embodiment, the final product comprises from 40 to 85% by weight of the base powder of the invention and from 15 to 60% by weight of postdosed ingredients of high bulk density and small particle size, for example from 15 to 35% by weight of sodium sulphate and from 0 to 45% of other postdosed ingredients.

In order to obtain a very high bulk density product, the granulometry of the postdosed material is matched to that of the base powder, the relationship being expressed in terms of the Rosin-Rammler particle size distribution described by Rosin and Rammler, J Inst Fuel 7 29-36 (1933). The postdosed solid material should have an overall Rosin-Rammler average particle size not exceeding 75%, and preferably not exceeding 70%, of that of the base powder. Typically the Rosin-Rammler average particle size of the base powder may be from 350 to 800 µm, and that of the postdosed material from 200 to 400 µm.

The postdosed solid material should also itself be of high bulk density: at least 850 g/litre, preferably at least 900 g/litre. These figures apply to the totality of postdosed solid material, that is to say, to a mixture of all the postdosed solids in the proportions in which they are to be present in the final product. In reality, of course, the various solids are likely to be dosed separately to the base powder, and some will have higher bulk densities than the overall figure while others will have lower bulk densities.

Preferably, the postdosed solid material contains a substantial proportion of sodium sulphate. This material may be obtained with a bulk density of 1200 g/litre or more, preferably at least 1300 g/litre, and a suitable particle size distribution. The use of very high bulk density sodium sulphate allows greater flexibility in the choice of any other solid postdosed ingredients.

In general, the man skilled in the art will have no difficulty in preparing sodium sulphate and other materials of suitable granulometry for postdosing. Commercial materials of large particle size can be ground and sieved while very fine materials can be compacted and sieved.

It should be emphasised that the presence of sodium sulphate is not essential in order to obtain high bulk density detergent compositions utilising the spray-dried base powder of the present invention, but it represents one possible approach.

Matching of the granulometry of the postdosed solid material to that of the base powder will give the maximum possible increase in bulk density as a result of the postdosing operation. A detergent composition produced by the process of the invention will have a particle size distribution such that the larger particles are predominantly derived from the spray-dried base powder while the smaller particles are predominantly derived from the postdosed solid materials, including sodium sulphate.

This embodiment of the invention offers another important benefit in addition to increased bulk density. Powders composed of a relatively coarse base powder and relatively fine postdosed material have surprisingly been found to exhibit significantly better dispensing properties in the washing machine, as compared with powders having a similar particle size distribution but composed of a relatively fine base powder and relatively coarse postdosed material.

In this context, it is strongly preferred that the postdosed material should not contain too high a level of very small particles or "fines": the content of particles smaller than 125 µm is preferably less than 15% by weight, and more preferably less than 10% by weight. The "fines" content of the base powder is also preferably less than 15% by weight.

These very high bulk density detergent compositions represent one especially preferred embodiment of the present invention. It is also within the scope of the invention, of course, to postdose to the base powder of the invention other materials outside the definitions given above, for example, larger particle size salts, to give detergent compositions having any desired properties.

Also within the scope of the invention are compositions in which the spray-dried powder of the invention is only a minor component, constituting for example from 10 to 30% by weight of the total product or possibly even less, and perhaps better regarded as an "adjunct" rather than as a base powder.

EXAMPLES

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

EXAMPLES 1 TO 16

Spray-dried base powders were prepared to the ocmpositions set out in the following Tables IA, IIA, IIIA and IVA, and the properties of these powders are given in the corresponding Tables IB, IIB, IIIB and IVB. Examples identified by number are within the invention, while examples identified by letter are comparative.

The polymer used in all the compositions was Sokalan (Trade Mark) CP5 ex BASF, an acrylic/maleic copolymer in sodium salt form.

The minor ingredients referred to in the Tables included antiredeposition agent, fluorescer and EDTA.

Compositions 1 to 6 in Tables IA and IB all contained relatively high levels, above 14.5% by weight, of anionic surfactant, and in every case the weight ratio of sodium carbonate to anionic surfactant was less than 1.1:1. It will be seen that at total electrolyte levels ranging from zero (insignificant) to 12.6% by weight, powders having particle porosites below 0.40 were obtained. The bulk density in every case exceeded 530 g/l. Although in some cases high moisture content led to rather high compressibility values, the other powder properties were excellent.

Compositions 7 to 11 in Tables IIA and IIB form a similar series but with lower levels, below 14.5% by weight, of anionic surfactant. The powders all had particle porosities below 0.4 even though in some cases the ratio of sodium carbonate to anionic surfactant exceeded 1.1:1.

It will be seen that Compositions 1 to 4 and 6 to 11 contained no significant amounts of any electrolyte other than sodium carbonate, while Composition 5 contained sodium carbonate and sodium silicate.

Composition 1 containing no sodium carbonate displayed a slight yellow discolouration, while the powders of Examples 2-11 were white.

Tables IIIA and IIIB show some compositions outside the invention. Comparative composition A contained just above 14.5% by weight of anionic surfactant, and the ratio of sodium carbonate to anionic surfactant was just above the limit of 1.1:1, and it will be seen that its particle porosity was 0.41: it should be compared with Example 10 which had the same ratio of sodium carbonate to anionic surfactant but a lower level (just below 14.5% by weight) of anionic surfactant, and a particle porosity of 0.36. Comparative Examples B, C and E show that higher levels of sodium carbonate, and higher ratios, gave even higher particle porosities. Comparative Example D shows the detrimental effect of including sodium sulphate.

Compositions 12 to 16 in Table IVA and IVB illustrate the invention in a series of compositions containing relatively higher proportions of anionic surfactant. The very high bulk densities of these powders will be noted. As with the earlier Examples, Composition 12 containing no sodium carbonate displayed a slight yellow discolouration, while the other powders were all white.

Table IA - Examples 1-6

	1	2	3	4	5	6
Linear alkylbenzene sulphonate (Na salt)	19.9	17.1	17.5	19.0	16.6	16.4
Nonionic surfactant	2.2	1.9	1.9	2.1	1.9	1.8
Zeolite	48.6	45.6	46.7	46.5	44.3	40.3
Polymer	8.8	7.6	7.8	8.4	7.4	7.2
Sodium carbonate	-	3.8	3.9	4.2	3.7	12.6
Sodium silicate	-	-	-	-	4.8	-
Minor ingredients	2.2	1.7	1.7	2.1	1.6	1.8
Moisture	18.3	22.3	20.5	17.7	19.7	19.9
Total electrolyte	0	3.8	3.9	4.2	8.5	12.6
Ratio of sodium carbonate to anionic surfactant	0	0.22	0.22	0.22	0.51	0.78

Table IB - Examples 1-6

	1	2	3	4	5	6
Relative humidity (%)	44.2	57.5	50.4	41.1	45.6	53.9
Particle porosity	0.31	0.32	0.24	0.30	0.34	0.38
Bulk density (g/l)	580	550	540	540	534	535
Dynamic flow rate (ml/s)	120	105	108	117	111	118
Compressibility (% v/v)	14	33	26	13	15	34
Agglomerate strength (N/cm^2)	22	14	36	42	40	12

Table IIA - Examples 7-11

	7	8	9	10	11
Linear alkylbenzene sulphonate (Na salt)	12.3	6.7	9.6	14.3	14.0
Nonionic surfactant	1.9	2.1	11.3	1.6	1.6
Soap	-	-	1.6	-	-
Zeolite	46.1	50.5	38.7	38.1	37.4
Polymer	7.6	8.4	6.5	6.4	6.2
Sodium carbonate	6.9	8.0	8.1	15.9	18.7
Minor ingredients	1.7	1.9	1.5	2.9	1.4
Moisture	23.5	22.4	22.7	20.8	20.7
Total electrolyte	6.9	8.0	8.1	15.9	18.7
Ratio of sodium carbonate to anionic surfactant	0.40	1.19	0.71	1.11	1.33

Table IIB - Examples 7-11

	7	8	9	10	11
Relative humidity (%)	46.8	48.0	59.5	58.4	58.1
Particle porosity	0.37	0.33	0.37	0.36	0.37
Bulk density (g/l)	455	535	497	515	520
Dynamic flow rate (ml/s)	114	110	103	117	122
Compressibility (% v/v)	24	11	32	32	27
Agglomerate strength (N/cm²)	59	93	8	8	11

Table IIIA - Comparative Examples A-E

	A	B	C	D	E
Linear alkylbenzene sulphonate (Na salt)	14.6	15.5	12.7	12.5	11.0
Nonionic surfactant	1.6	1.7	1.4	1.4	1.2
Zeolite	38.9	37.8	33.9	33.2	29.3
Polymer	6.5	6.9	5.6	5.5	4.9
Sodium carbonate	16.0	20.6	28.2	2.8	33.9
Sodium sulphate	-	-	-	27.0	-
Minor ingredients	3.0	1.7	1.2	1.1	1.1
Moisture	20.4	15.8	16.9	16.4	18.6
Total electrolyte	16.0	20.6	28.2	29.8	33.9
Ratio of sodium carbonate to anionic surfactant	1.11	1.33	2.22	0.22	3.11

Table IIIB - Comparative Examples A-E

	A	B	C	D	E
Relative humidity (%)	53.9	48.0	50.4	56.0	55.8
Particle porosity	0.41	0.51	0.43	0.55	0.53
Bulk density (g/l)	490	440	531	445	445
Dynamic flow rate (ml/s)	112	104	117	90	116
Compressibility (% v/v)	34	19	10	30	23
Agglomerate strength (N/cm²)	7	15	18	5	9.5

Table IVA - Examples 12-16

	12	13	14	15	16
Linear alkylbenzene sulphonate (Na salt)	16.2	15.3	13.7	14.2	12.8
Nonionic surfactant	1.8	1.7	1.5	1.6	1.4
Zeolite	53.9	50.9	45.6	47.4	49.9
Polymer	5.4	5.1	3.0	4.8	2.9
Sodium carbonate	-	3.4	10.6	11.1	10.1
Minor ingredients	1.8	1.5	1.4	1.4	1.3
Moisture	21.9	22.1	24.2	19.5	21.6
Total electrolyte	0	3.4	10.6	11.1	10.1
Ratio of sodium carbonate to anionic surfactant	0	0.22	0.78	0.78	0.78

Table IVB - Examples 12-16

	12	13	14	15	16
Relative humidity (%)	55.4	55.8	57.5	53.6	55.8
Particle porosity	0.25	0.26	0.31	0.34	0.40
Bulk density (g/l)	590	595	525	560	590
Dynamic flow rate (ml/s)	106	111	122	118	100
Compressibility (% v/v)	29	21	25	22	29
Agglomerate strength (N/cm^2)	42	34	12	25	11

EXAMPLES 17 AND 18

Fully formulated detergent compositions were prepared by postdosing various ingredients, as specified below, to the base powders 4, B and 13 described above.

	17(4)	F(B)	18(13)
Base powder			
Linear alkylbenzene sulphonate	9.0	9.0	9.0
Nonionic surfactant	1.0	1.0	1.0
Zeolite	22.0	22.0	30.0
Polymer	4.0	4.0	3.0
Sodium carbonate	2.0	12.0	2.0
Antiredeposition agent, fluorescer, EDTA etc.	1.0	1.0	1.0
Moisture	8.4	9.2	10.6
Total base powder	47.4	58.2	56.6

<u>Postdosed material</u>

Nonionic surfactant	3.0	3.0	3.0
Sodium sulphate	29.9	16.0	21.5
Sodium carbonate	1.9	-	1.0
Sodium silicate A.1	5.0	5.0	5.0
Bleach ingredients (sodium perborate, TAED, stabiliser) and minor ingredients (enzyme, lather suppressor, perfume)	12.8	17.8	12.9
	100.0	100.0	100.0

The properties of the powders were as follows:

	17	F	18
Rosin-Rammler average particle sizes:			
base powder (µm)	420	360	380
postdosed material (µm)	315	330	320
sodium sulphate (µm)	290	290	290
postdosed material as % of base powder	75	92	84
final powder	360	335	335
Bulk densities (g/litre):			
base powder	560	440	595
postdosed material	930	720	930
sodium sulphate	1600	1600	1600
final powder	740	580	740
increase on postdosing	180	140	145
Fines (% particles < 125 µm):	11	4	12
Dynamic flow rate (ml/s)	87	83	86

Composition 17 was a powder in accordance with an especially preferred embodiment of the invention since the postdosed material had an overall Rosin-Rammler average particle size (315 µm) only 75% of that of the base powder, and was also of high bulk density (930 g/litre). An increase of bulk density of 180 g/litre to the very high figure of 740 g/litre was thereby achieved.

Composition 18 is an example of a powder in which the base powder was of exceptionally high bulk density (595 g/litre). The postdosed material was also of very high bulk density (930 g/litre) but of larger particle size (320 µm, 84%) in relation to the base powder (380 µm) so the increase achieved by postdosing (145 g/litre) was smaller than in Composition 17. However, because of the exceptionally high bulk density of the base powder the bulk density of the final powder was just as high (740 g/litre) as that of Composition 17.

Comparative Composition F was composed of a base powder of low bulk density (440 g/litre) and postdosed material of relatively low bulk density (720 g/litre) and a particle size not significantly smaller than that of the base powder. Postdosing achieved a bulk density increase of 140 g/litre, but the final figure (580 g/litre) was considerably lower than the preferred target figure of 650 g/litre.

Claims

1. A spray-dried zero-phosphate detergent powder comprising:
(a) from 5 to 60% by weight, in total, of one or more anionic detergent-active compounds;
(b) optionally from 0 to 30% by weight of one or more nonionic detergent-active compounds;
(c) from 15 to 86% by weight of crystalline or amorphous sodium aluminosilicate builder;
(d) from 2 to 40% by weight of a polymeric polycarboxylate;
(e) optionally sodium carbonate;
(f) optionally other salts;
(g) optionally conventional minor ingredients;
characterised in that the powder has a total electrolyte level not exceeding 25% by weight and a particle porosity not exceeding 0.40, and if the amount of anionic detergent-active compound (a) exceeds 14.5% by weight the weight ratio of sodium carbonate (e) to anionic detergent-active compound (a) does not exceed 1.1:1.

2. A powder as claimed in claim 1, characterised in that the total electrolyte level does not exceed 20% by weight.

3. A powder as claimed in claim 2, characterised in that the total electrolyte level does not exceed 15% by weight.

4. A powder as claimed in claim 3, characterised in that the total electrolyte level does not exceed 13% by weight.

5. A powder as claimed in any preceding claim, characterised in that it comprises from 2 to 20% by weight of sodium carbonate (e).

6. A powder as claimed in claim 5, characterised in that it comprises from 5 to 15% by weight of sodium carbonate (e).

7. A powder as claimed in any preceding claim, characterised in that it comprises sodium carbonate (e) in a weight ratio to the anionic surfactant (a) within the range of from 0.1:1 to 0.8:1.

8. A powder as claimed in any preceding claim, characterised by a particle porosity of less than 0.35.

9. A powder as claimed in any one of claims 1 to 8, characterised in that it is substantially free of electrolytes other than sodium carbonate (e).

10. A powder as claimed in any one of claims 1 to 8, characterised in that it comprises as a salt (f) sodium silicate in an amount not exceeding 10% by weight.

11. A powder as claimed in claim 10, characterised in that the amount of sodium silicate does not exceed 5% by weight.

12. A powder as claimed in claim 10 or claim 11, characterised in that it is substantially free of electrolytes other than sodium carbonate (e) and sodium silicate (f).

13. A powder as claimed in any preceding claim, characterised in that it comprises from 10 to 60% by weight, in total, of anionic detergent-active compounds (a).

14. A powder as claimed in any preceding claim, characterised in that a nonionic detergent-active compound (b) is present, the weight ratio of anionic detergent-active compound (a) to nonionic detergent-active compound (b) being at least 0.67:1.

15. A powder as claimed in claim 14, characterised in that the weight ratio of anionic detergent-active compound to nonionic detergent-active compound is within the range of from 1:1 to 10:1.

16. A powder as claimed in any preceding claim, characterised by a relative humidity not exceeding 70%.

17. A powder as claimed in claim 16, characterised by a relative humidity within the range of from 45 to 65% by weight.

18. A process for the preparation of a zero-phosphate detergent powder as claimed in any preceding claim, which comprises spray-drying an aqueous slurry to form a powder having a particle porosity not exceeding 0.40, the slurry comprising:
(a) one ore more anionic detergent-active compounds, in an amount of from 5 to 60% by weight based on the powder,

(b) optionally one or more nonionic detergent-active compounds, in an amount of from 0 to 30% by weight based on the powder,

(c) a crystalline or amorphous sodium aluminosilicate builder, in an amount of from 15 to 86% by weight based on the powder,

(d) a polymeric polycarboxylate, in an amount of from 2 to 40% by weight based on the final powder,

(e) optionally sodium carbonate,

(f) optionally other salts,

(g) optionally conventional minor ingredients,

characterised in that the total electrolyte level in the slurry is such that the total electrolyte level of the powder does not exceed 25% by weight, and in that if the amount of anionic detergent-active compound (a) exceeds 14.5% by weight the weight ratio of sodium carbonate (e) to anionic detergent-active compound (a) does not exceed 1.1:1.

19. A granular zero-phosphate detergent composition characterised in that it comprises:

(i) from 10 to 100% by weight of a spray-dried powder as claimed in any one of claims 1 to 16,

(ii) from 0 to 90% by weight of one or more postdosed solid and/or liquid ingredients.

20. A composition as claimed in claim 19, characterised in that it comprises:

(i) from 30 to 90% by weight of the spray-dried powder, and

(ii) from 10 to 70% by weight of postdosed ingredients.

21. A composition as claimed in claim 20, characterised in that it comprises:

(i) from 40 to 85% by weight of the spray-dried powder, and

(ii) from 15 to 60% by weight of postdosed ingredients.

22. A composition as claimed in claim 21, characterised in that it comprises:

(i) from 40 to 85% by weight of the spray-dried powder,

(ii) from 15 to 35% by weight of postdosed sodium sulphate, and

(iii) optionally from 0 to 45% by weight of other postdosed material.

23. A composition as claimed in claim 21 or claim 22, characterised in that any solid postdosed material has an overall Rosin-Rammler average particle size not exceeding 75% of the Rosin-Rammler average particle size of the spray-dried powder (i).

24. A composition as claimed in any one of claims 21 to 23, characterised by a bulk density of at least 650 g/litre.