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

Waschmittelpulver und Verfahren zu deren Herstellung

Compositions détergentes en poudre et leur procédé de préparation

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(56) References cited:
EP-A- 0 045 208 **EP-A- 0 206 522**
DE-A- 285 155 **DE-A- 3 400 008**
FR-A- 2 257 680 **US-A- 2 423 450**

• **Robert N.Wenzel, "Resistance of solid surfaces to wetting by water", Ind.Eng.Chem., vol.28,no.8, 1936, pages 988-994**
• **Judson L.Ihrig, David Y.F.Lai, "Contact angle measurements", J. Chem. Educ., vol. 34, no. 4, 1957, pages 196-198**
• **"Commercial Waxes", ed. H. Bennett, Chemical Publishing Co., New York, 2.Edition, 1956, page 193**
• **H.Bennett, "Industrial waxes", Chem. Publishing Co., New York, 1975, page 89**

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DescriptionTECHNICAL FIELD

5 **[0001]** The present invention relates to detergent powders prepared at least in part by spray-drying and intended for use in drum-type front-loading washing machines. The invention is of especial applicability to powders containing no, or reduced levels of, phosphate builders and to powders of high bulk density.

BACKGROUND

10 **[0002]** In recent years the trend in detergent compositions has been towards reducing or eliminating phosphate builders. The replacement of sodium tripolyphosphate as a builder in detergent powders by its most popular zero-phosphate substitute, crystalline sodium aluminosilicate (zeolite), has led to a number of difficulties with the structure and physical properties of the powder. One such problem that has been encountered is the tendency of zeolite-built
15 powders to dispense less well in front-loading automatic washing machines than do their phosphate-built counterparts; a higher proportion of the powder dosed into the machine is left behind in the dispenser, leading to product wastage and clogging. This problem is especially marked at low wash temperatures.

[0003] The tendency towards poor dispensing has been exacerbated by the recent trend in the detergents industry towards higher bulk density powders.

20 **[0004]** We have now found that the dispensing behaviour of those powders that are especially prone to this problem may be substantially improved by including in the powders low levels of certain paraffin waxes, either via the slurry or by subsequent spray-on.

[0005] Surprisingly, the incorporation of the materials via the slurry also improves powder structure and raises the
25 bulk density.

DEFINITION OF THE INVENTION

[0006] In a first aspect, the invention comprises a spray-dried detergent powder as defined by claim 1.

30 **[0007]** In a second aspect, the invention provides a process for the preparation of a detergent powder, the process being as defined by claim 7.

PRIOR ART

35 **[0008]** FR-A-2 257 680 discloses phosphate containing granular detergent compositions which include a microcrystalline paraffin wax as a foam control agent. It is generally mentioned in this document that suitable waxes appear to be those which can be solubilized by the surfactant employed. Further examples of detergent compositions containing foam control systems that include hydrocarbon waxes are given in EP-A-87 233, EP-A-109 247 and EP-A-206 522 (Unilever); GB 1 492 939, GB-B-2 040 982, EP-A-8 829, EP-A-8 830 and JP-A-56 034797 (Procter & Gamble);
40 DE-A-3 436 194, US 4 590 194 and EP-A-150 386 (Henkel). US 4 196 104 (Procter & Gamble) discloses spraying an antistatic composition comprising a quaternary ammonium compound and a paraffin wax (as dispersion inhibitor) onto detergent base granules.

DETAILED DESCRIPTION OF THE INVENTION

45 **[0009]** The first aspect of the invention is a detergent powder prepared at least in part by spray-drying. The composition of the invention may be a fully formulated detergent composition prepared wholly by spray-drying; a spray-dried base to which other ingredients may be admixed to form a finished product; or a finished product of that type, comprising a spray-dried base in admixture with other ingredients.

50 **[0010]** As essential ingredients, the detergent powder of the invention contains at least 5% by weight of one or more anionic and/or nonionic surfactants, from 20 to 80% by weight of crystalline or amorphous aluminosilicate detergency builder, and a specified paraffin wax that influences its dispensing behaviour in a particular manner.

[0011] The composition of the invention may also contain any of the materials conventionally included in detergent compositions. These are described in more detail below.

55 The surfactant component

[0012] The total amount of surfactant present in the composition of the invention will generally range from 5 to 40% by weight, more preferably from 10 to 30% by weight and especially from 12 to 20% by weight. These figures are

typical for fully formulated detergent compositions, and where a spray-dried base forms only part of such a composition the surfactant content of that base, as a percentage, may of course be higher.

[0013] The invention relates to compositions containing anionic surfactant. When such powders are prepared by spray-drying, the high-foaming anionic surfactant tends to cause "puffing" (entrainment of air) in the slurry, so that highly porous particles are formed in the spray-drying tower. These particles may be very crisp and free-flowing, but may be of lower bulk density than desired. Surprisingly, the incorporation in the slurry of a paraffin wax, in accordance with the present invention, has been found to result in an increase in bulk density. The amount of anionic surfactant present is at least 5% by weight, and may suitably be in the range of from 5 to 30% by weight, preferably from 5 to 10% by weight, these figures again being based on a fully formulated detergent composition.

[0014] Anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅; primary and secondary alkyl sulphates, particularly sodium C₁₂-C₁₅ primary alcohol sulphates; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

[0015] Preferably, the composition of the invention also contains one or more nonionic surfactants. Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C₁₂-C₁₅ primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.

[0016] The weight ratio of anionic surfactant to nonionic surfactant is preferably at least 0.67:1, more preferably at least 1:1, and most preferably within the range of from 1:1 to 10:1, in order to obtain the optimum detergency and foaming properties appropriate for front-loading automatic washing machines. These ratios of course apply to fully formulated products. A spray-dried base that is to form only part of a product may contain a lower proportion of, or no, nonionic surfactant, the balance of the nonionic surfactant being added after the spray-drying tower.

The paraffin wax

[0017] The characterising feature of the composition of the present invention is the presence of a low level (0.1-6.0% by weight) of a paraffin wax. The hydrophobicity of this material is expressed in terms of its contact angle to water, which must be at least 75°, preferably at least 85°.

[0018] The melting point of the paraffin wax is also important: it appears that the wax should be solid at ambient temperature but liquid at slurry-processing temperature (generally in the range of from 60 to 100°C). The melting point of the paraffin wax should lie within the range of from 30 to 100°C, preferably from 40 to 80°C, more preferably within the range of from 50 to 60°C. Paraffin oil liquid at ambient temperature has been found to be ineffective in the context of the present invention, and also tends to give wet, sticky powders with unacceptable flow properties.

[0019] It is also essential that the paraffin wax be substantially insoluble in anionic and nonionic surfactants: the microcrystalline waxes of FR-A-2 257 680 and GB 1 492 939 (Procter & Gamble) are stated to be chosen for their solubility in the surfactant present, and are unsuitable for use in the present invention for that reason as well as for the reason that their melting points are too high.

[0020] The paraffin wax is preferably present in an amount of from 0.2 to 4% by weight. In a fully formulated product the optimum level for paraffin wax appears to be from 0.3 to 2% by weight.

Incorporation of the paraffin wax

[0021] The composition of the invention is prepared by a process which includes the step of spray-drying an aqueous crutcher slurry. This slurry will normally contain all those desired ingredients sufficiently heat-stable to survive the spray-drying process, notably anionic surfactants, builders, inorganic salts, sodium silicate, Polymers and fluorescers. More heat-sensitive ingredients can be postdosed to, or sprayed onto, the spray-dried base.

[0022] There are two methods by means of which the paraffin wax characteristic of the invention may be incorporated. It may be included in the slurry, in which case it is preferably used in an amount of from 0.2 to 4.0% by weight, more preferably from 0.3 to 2.0% by weight, based on the final powder including any postdosed ingredients. It may be advantageous to premix the paraffin wax with surfactant, especially nonionic surfactant, before admixture with other slurry ingredients.

[0023] It has surprisingly been found that incorporation of paraffin wax via the slurry not only improves dispensing behaviour, but also improves powder structure and, when anionic surfactant is present, also raises the bulk density. Flow properties are not detrimentally affected.

[0024] The second method by which the paraffin wax may be incorporated in a detergent composition of the invention is by spraying it in liquefied form onto the spray-dried powder. If the final product is to include postdosed solid ingredients, for example, sodium perborate, bleach activator granules, enzyme granules or antifoam granules, the paraffin wax should be sprayed on after addition of those ingredients so as to cover the whole powder.

[0025] If desired, the paraffin wax may be melted and sprayed directly onto the powder. The paraffin wax is then

preferably used in an amount of from 2.0 to 6.0% by weight, more preferably from 3.0 to 4.0% by weight.

[0026] According to a preferred embodiment of the invention, however, the paraffin wax is premixed with nonionic surfactant to form a coating composition which may then be sprayed onto the detergent powder. Lower levels of the paraffin wax, for example, 0.2 to 4.0%, preferably 0.3 to 2.0%, are then found to be effective. The coating composition consists essentially of 2-50% by weight of the paraffin wax and 50-98% by weight of nonionic surfactant. The coating composition desirably contains 8-15% by weight of paraffin wax and 85-92% by weight of nonionic surfactant. The paraffin wax, and if necessary the nonionic surfactant, are melted to form the coating composition, which is applied as a liquid, suitably at a temperature of 40-60 ° C.

[0027] The coating composition should be substantially free of other ingredients that might interfere with the beneficial effect of the paraffin wax on dispensing.

Dispensing behaviour

[0028] It is an essential feature of the detergent powder of the invention that the incorporation as specified above of a paraffin wax should bring about an improvement in dispensing behaviour. Dispensing is assessed by means of a standard test using a Philips (Trade Mark) AWB 126/7 washing machine using a 100 g powder dose and a water fill of 5 litres at 20°C flowing in over a period of 1 minute. The dry weight of powder remaining in the dispenser, in grams, then represents the weight percentage of powder not dispensed into the machine (the residue). It will be appreciated that this test is stringent, using a low water inlet temperature and flow rate, and a machine with a drawer-type dispenser which is particularly vulnerable to high residues and clogging. According to the invention, the incorporation of the paraffin wax should effect a reduction in the residue of at least 10 percentage points, preferably at least 20 percentage points. For the purposes of comparison, a powder having essentially the same composition but with an inorganic salt (sodium carbonate or sodium sulphate) to make up the difference should be used.

[0029] Clearly an improvement of this magnitude can only be observed if the control powder exhibits poor dispensing properties. The present invention is therefore especially applicable to powders which, without the paraffin wax, give dispenser residues of at least 10%, more especially at least 20%, by weight. That is especially likely to be the case if the powder is a zero-phosphate composition built with crystalline (zeolite) or amorphous sodium aluminosilicate; if it contains less than 10% sodium silicate; or if it has a bulk density of 450 g/litre or more, preferably 600 g/litre or more.

Optional components

[0030] As indicated previously, the detergent powder of the invention can contain any of the ingredients conventionally present in compositions intended for the washing of fabrics in front-loading automatic washing machines. Examples of such components include inorganic and organic detergency builders, other inorganic salts, bleaches, fluorescers, polymers, lather control agents, enzymes and perfumes.

[0031] If desired, the powder of the invention may contain one or more soaps of fatty acids, in addition to the non-soap anionic surfactant mentioned above.

[0032] The powder will also contain one or more detergency builders. The invention is of especial applicability to zero-phosphate powders containing from 20 to 80 % by weight of crystalline (zeolite) or amorphous aluminosilicate. Other supplementary, builders may also be present, for example, polycarboxylate polymers such as polyacrylates, acrylic-maleic copolymers, or acrylic phosphinates; monomeric polycarboxylates such as nitrilotriacetates and ethylene diamine tetraacetates; inorganic salts such as sodium carbonate; and many other materials familiar to the skilled detergent formulator.

[0033] If desired, the powder of the invention may contain sodium silicate. High levels of silicate can in themselves have a beneficial effect on dispensing, as well as on powder structure and prevention of machine corrosion, but are undesirable in powders containing aluminosilicate because the two components react together to form insoluble siliceous species. The present invention enables the dispensing behaviour of zeolite-built powders to be improved without a corresponding increase in the level of insoluble material. Accordingly, the invention relates to powders containing less than 10% by weight, more especially less than 5% by weight, of sodium silicate.

[0034] Other materials that may be present in the powder of the invention include fluorescers, antiredeposition agents, inorganic salts such as sodium sulphate, enzymes, lather control agents, bleaches, bleach activators, and bleach stabilisers. These may be included in the spray-dried base powder or postdosed according to their known suitability for undergoing spray-drying processes and their compatibility with other slurry ingredients.

[0035] The invention is further illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated.

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EXAMPLES

Examples 1 to 4

5 **[0036]** Zero-phosphate detergent base powders containing various amounts of paraffin wax (melting point 52-54°C) or paraffin oil were prepared, by slurry-making and spray-drying, to the following nominal composition:

	Parts
Sodium linear alkylbenzene sulphonate	9.0
Nonionic surfactant	1.0
Zeolite (anhydrous)	24.0
Acrylic/maleic copolymer*	4.0
Sodium carbonate	2.0
Minor ingredients	0.83
Moisture	10.0
Paraffin wax or paraffin oil	0, 0.25, 0.5, 1.0, 2.0 1.0
Total	51-53 parts

*Sokalan (Trade Mark) CP5 ex BASF.

[0037] To the base powder were postdosed the following ingredients:

	Parts
Burkeite/nonionic surfactant adjunct*	13.0
Sodium perborate monohydrate	8.0
TAED granules	3.0
Dequest	0.2
Enzyme granules	0.6
Lather control granules	3.0
Sodium alkaline silicate	5.0
Sodium carbonate	5.4
Sodium sulphate	to 100.0

*as described and claimed in EP 221 776A (Unilever) 2

[0038] The following table shows the six compositions prepared, their dispenser residues measured in accordance with the defined test given above, and their powder properties:

	A	1	2	3	4	B
Paraffin wax	0	0.25	0.5	1.0	2.0	0
Paraffin oil	0	0	0	0	0	1.0
Sodium sulphate	11.65	11.32	10.87	10.62	9.82	10.62
Dispenser residue	42	20	0	0	0	33
Bulk density (g/l)	680	609	755	724	685	
Dynamic flow rate	109	111	109	109	90	
Compressibility (%)	34	16	24	25	21	

[0039] It will be noted that even the low level of 0.25 parts of paraffin wax effected a substantial improvement in dispensing behaviour, and no dispenser residues at all were observed when higher levels of paraffin wax were used. The higher levels also brought about increases in bulk density, and improvements in powder structure (compressibility) were observed at all levels.

[0040] The dispensing and powder properties of the spray-dried base powders of Examples 2-4 and Comparative

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Example A were also examined. The full formulations of these base powders were as follows:

	A	2	3	4
Sodium LAS	17.96	17.67	17.59	17.30
Nonionic surfactant	1.99	1.96	1.95	1.92
Zeolite (anhydrous)	47.89	47.13	46.89	46.17
Copolymer	7.89	7.85	7.82	7.70
Sodium carbonate	3.99	3.93	3.91	3.85
Minor ingredients	1.66	1.63	1.62	1.60
Moisture	18.53	18.85	18.27	17.61
Paraffin wax	0	0.98	1.9	3.85
Total	100.0	100.0	100.0	100.0

[0041] The full formulation of the base powder of Comparative Example B was identical to that of Example 3, except that the paraffin wax was replaced by paraffin oil. The dispensing residues of the base powders, determined by the test described above, and their powder properties were as follows:

	A	2	3	4
Dispenser residue (%)	46	0	0	0
Bulk density (g/l)	540	585	525	586
DFR (ml/s)	108	115	117	109
Compressibility (% v/v)	36	29	31	30

[0042] The dispenser residue of the base powder of Comparative Example B was 15%.

Example 5

[0043] Zero-phosphate detergent powders similar to those of Examples 1-4 but containing a higher level of anionic surfactant were prepared, to the following formulations (in weight %):

Spray-dried base powder	C	5
Sodium linear alkylbenzene sulphonate	11.1	11.1
Nonionic surfactant	1.2	1.2
Zeolite (anhydrous)	24.0	24.0
Acrylic/maleic copolymer*	4.0	4.0
Sodium carbonate	2.0	2.0
Sodium alkaline silicate	0.5	0.5
Sodium sulphate	1.3	1.3
Minor ingredients	0.83	0.83
Paraffin wax (m.pt. 52-54°C)	0	1.0
Moisture	9.50	9.95

*Sokalan (Trade Mark) CP5 ex BASF.

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Postdosed ingredients		
Burkeite/nonionic surfactant adjunct	16.09	16.09
Sodium perborate monohydrate	8.0	8.0
TAED granules	3.0	3.0
Dequest	0.75	0.75
Enzyme granules	0.5	0.5
Lather control granules	2.4	2.4
Sodium carbonate	3.27	3.27
Sodium sulphate	11.43	9.98
Total	100.0	100.0

[0044] The dispensing and powder properties of these products were as follows:

	C	5
Dispenser residue (%):		
of base	80	0
of whole product	55	3
Bulk density (g/l)	660	648
Dynamic flow rate (ml/s)	91	97
Compressibility (% v/v)	10	17

[0045] With this product, which already exhibited a very high bulk density and excellent powder properties, no further improvement in these respects resulted from the inclusion of paraffin wax in the base powder. A marked improvement in dispensing behaviour was, however, observed.

Examples 6 to 8

[0046] A zero-phosphate detergent base powder similar to that of Comparative Example A was prepared, by slurry-making and spray-drying, to the following formulation:

	Parts	%
Sodium linear alkylbenzene sulphonate	9.0	18.37
Nonionic surfactant	1.0	2.04
Zeolite (anhydrous)	24.0	48.98
Acrylic/maleic copolymer*	4.0	8.16
Sodium carbonate	2.0	4.08
Minor ingredients	0.83	1.69
Moisture	8.17	16.68

*Sokalan (Trade Mark) CP5 ex BASF.

[0047] Samples of this base powder were post-sprayed with molten paraffin wax (melting point 52-54°C) in various amounts, as shown in the table below. The dispensing properties of the samples, and of the base powder without wax spray-on (Comparative Example D), are also shown in the table.

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	D	6	7	8
Paraffin wax sprayed on:				
parts	0	0.3	1.0	2.0
%	0	0.61	2.0	3.92
Dispenser residue (%)	37	12	10	1

[0048] It will be seen that the sprayed-on paraffin wax effected a substantial reduction in dispenser residues: at the 2 parts (3.92%) level these were negligibly low.

Examples 9 to 11

[0049] The procedure of Examples 6 to 8 was repeated, but this time the paraffin wax was melted and dispersed in liquid nonionic surfactant, and the coating composition thus obtained was sprayed onto the base powder. The three coating compositions used were as follows, the parts being on the same basis as those of the base powder D, and the percentages being based on the coating composition:

	Parts	%
Example 9:		
Paraffin wax	0.3	9.1
Nonionic surfactant	3.0	90.9
Example 10:		
Paraffin wax	1.0	25.0
Nonionic surfactant	3.0	75.0
Example 11:		
Paraffin wax	2.0	40.0
Nonionic surfactant	3.0	60.0

[0050] Dispensing and powder properties were as follows:

	D	9	10	11
Wax/nonionic spray-on:				
Total (parts)	0	3.3	4.0	5.0
Total (% on base powder)	0	6.31	7.55	9.26
Dispenser residue (%)	37	0	0	0
Bulk density (g/ml)	450	512		
Dynamic flow rate (ml/s)	108	105		
Compressibility (% v/v)	27	21		

[0051] Comparison with Examples 6 to 8 shows that when the paraffin wax was pre-dispersed in nonionic surfactant it was effective at a lower level in improving dispensing behaviour. Bulk density and compressibility were improved even at the low level of 0.3 parts (0.61%) of paraffin wax.

Claims

1. A spray-dried detergent powder being substantially free from inorganic phosphate and comprising at least 5% by weight of one or more anionic surfactants, from 20 to 80% by weight of crystalline or amorphous aluminosilicate detergency builder, no more than 10% by weight of alkali metal silicate, and from 0.1 to 6.0% by weight of a paraffin wax which is water-insoluble and substantially insoluble in anionic and nonionic surfactants and has a melting point within the range of from 30 to 100°C and a contact angle to water of at least 75°, **characterised in that** the powder is obtainable by a process including the step of spray-drying an aqueous slurry of detergent ingredients,

the paraffin wax being either incorporated in the slurry or sprayed onto the spray-dried powder and **in that** the powder is substantially free of paraffin oil.

- 5
2. A powder as claimed in claim 1, **characterised in that** the paraffin wax has a contact angle to water of at least 85°.
3. A powder as claimed in claim 1 or claim 2, **characterised in that** the paraffin wax has a melting point within the range of from 40 to 80°C.
- 10
4. A powder as claimed in any preceding claim, **characterised in that** the amount of paraffin wax incorporated in the powder is within the range of from 0.2 to 4.0% by weight.
5. A powder as claimed in any preceding claim, **characterised in that** it has a bulk density of at least 450 g/litre.
- 15
6. A powder as claimed in claim 5, **characterised in that** it has a bulk density of at least 600 g/litre.
7. A process for the preparation of a detergent powder being substantially free from inorganic phosphate and comprising at least 5% by weight of one or more anionic surfactants, from 20 to 80% by weight of crystalline or amorphous aluminosilicate detergency builder no more than 10% by weight of alkalimetal silicate and 0.1 to 6.0% by weight based on the powder of a paraffin wax which is water-insoluble and substantially insoluble in anionic and nonionic surfactants and has a melting point within the range of from 30 to 100°C and a contact angle to water of at least 75°, the process including the step of spray-drying an aqueous slurry, **characterised in that** the paraffin wax is either incorporated in the slurry or sprayed onto the spray-dried powder and **in that** the powder is substantially free of paraffin oil.
- 20
8. A process as claimed in claim 7, **characterised in that** the paraffin wax is incorporated via the slurry, in an amount of from 0.2 to 4.0% by weight based on the powder.
- 25
9. A process as claimed in claim 8, **characterised in that** the paraffin wax is premixed with nonionic surfactant before admixture with other slurry ingredients.
- 30
10. A process as claimed in claim 7, **characterised in that** the paraffin wax in liquefied form is sprayed onto the spray-dried powder, in an amount of from 0.2 to 4.0% by weight on the powder thus obtained.
- 35
11. A process as claimed in claim 10, **characterised in that** it incorporates spraying onto the powder a coating composition consisting essentially of:
- (i) from 2 to 50% by weight of the paraffin wax dispersed in
- (ii) from 50 to 98% by weight of nonionic surfactant, the percentages being on the coating composition.
- 40
12. A process as claimed in claim 11, **characterised in that** the coating composition consists essentially of from 5 to 30% by weight of paraffin wax and from 70 to 95% by weight of nonionic surfactant.
- 45
13. A process as claimed in claim 12, **characterised in that** the coating composition consists essentially of from 8 to 15% by weight of paraffin wax and from 85 to 92% by weight of nonionic surfactant.

Patentansprüche

- 50
1. Sprühgetrocknetes Waschmittelpulver, das im wesentlichen frei von anorganischem Phosphat ist und wenigstens 5 Gew.% eines oder mehrerer anionischer oberflächenaktiver Mittel, von 20 bis 80 Gew.% eines kristallinen oder amorphen Aluminiumsilikatwaschkraftbuilders und nicht mehr als 10 Gew.% Alkalimetallsilikat und von 0.1 bis 6.0 Gew.% eines Paraffinwachses umfaßt, das wasserunlöslich und im wesentlichen in anionischen und nichtionischen oberflächenaktiven Mitteln unlöslich ist und einen Schmelzpunkt im Bereich von 30 bis 100°C und einen Benetzungswinkel zu Wasser von wenigstens 75° hat, **dadurch gekennzeichnet, daß** das Pulver erhältlich ist durch ein Verfahren, das den Schritt des Sprühtrocknens einer wäßrigen Aufschlämmung aus Waschmittelzutaten beinhaltet, wobei das Paraffinwachs entweder der Aufschlämmung beigemischt wird oder auf das sprühgetrocknete Pulver aufgesprüht wird, und dadurch, daß das Pulver im wesentlichen frei von Paraffinöl ist.
- 55

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2. Pulver nach Anspruch 1, **dadurch gekennzeichnet, daß** das Paraffinwachs einen Benetzungswinkel zu Wasser von wenigstens 85° hat.
- 5 3. Pulver nach Anspruch 1 oder Anspruch 2, **dadurch gekennzeichnet, daß** das Paraffinwachs einen Schmelzpunkt im Bereich von 40 bis 80°C hat.
4. Pulver nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, daß** die Menge des dem Pulver beigemengten Paraffinwachses im Bereich von 0.2 bis 4.0 Gew.% liegt.
- 10 5. Pulver nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet, daß** es eine Schüttdichte von wenigstens 450 g/Liter besitzt.
6. Pulver nach Anspruch 5, **dadurch gekennzeichnet, daß** es eine Schüttdichte von wenigstens 600 g/Liter besitzt.
- 15 7. Verfahren zur Herstellung eines Waschmittelpulvers, das im wesentlichen frei von anorganischem Phosphat ist und wenigstens 5 Gew.% eines oder mehrerer anionischer oberflächenaktiven Mittel, von 20 bis 80 Gew.% eines kristallinen oder amorphen Aluminiumsilikatwaschkraftbuilders und nicht mehr als 10 Gew.% Alkalimetallsilikat und von 0.1 bis 6.0 Gew.% bezogen auf das Pulver eines Paraffinwachses umfaßt, das wasserunlöslich und im wesentlichen in anionischen und nichtionischen oberflächenaktiven Mitteln unlöslich ist und einen Schmelzpunkt
20 im Bereich von 30 bis 100°C und einen Benetzungswinkel zu Wasser von zumindest 75° aufweist, wobei das Verfahren den Schritt des Sprühtrocknens einer wäßrigen Aufschlämmung beinhaltet, **dadurch gekennzeichnet, daß** das Paraffinwachs entweder der Aufschlämmung beigemengt wird oder auf das sprühgetrocknete Pulver gesprüht wird, und daß das Pulver im wesentlichen frei von Paraffinöl ist.
- 25 8. Verfahren nach Anspruch 7, **dadurch gekennzeichnet, daß** das Paraffinwachs über die Aufschlämmung in einer Menge von 0.2 bis 4.0 Gew.%, bezogen auf das Pulver, beigemengt wird.
9. Verfahren nach Anspruch 8, **dadurch gekennzeichnet, daß** das Paraffinwachs vor der Vermischung mit den
30 anderen Inhaltsstoffen der Aufschlämmung mit einem nichtionischen oberflächenaktiven Mittel vorgemischt wird.
10. Verfahren nach Anspruch 7, **dadurch gekennzeichnet, daß** das Paraffinwachs in verflüssigter Form auf das sprühgetrocknete Pulver in einer Menge von 0.2 bis 4.0 Gew.%, bezogen auf das so erhaltene Pulver, gesprüht wird.
- 35 11. Verfahren nach Anspruch 10, **dadurch gekennzeichnet, daß** es das Aufsprühen einer Beschichtungszusammensetzung auf das Pulver umfaßt, die im wesentlichen besteht aus:
- (i) von 2 bis 50 Gew.% Paraffinwachs, dispergiert in
40 (ii) von 50 bis 98 Gew.% nichtionischem oberflächenaktiven Mittel
- wobei die Prozentwerte auf die Beschichtungszusammensetzung bezogen sind.
12. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, daß** die Beschichtungszusammensetzung im wesentlichen aus von 5 bis 30 Gew.% Paraffinwachs und von 70 bis 95 Gew.% nichtionischem oberflächenaktiven Mittel
45 besteht.
13. Verfahren nach Anspruch 12, **dadurch gekennzeichnet, daß** die Beschichtungszusammensetzung im wesentlichen aus von 8 bis 15 Gew.% Paraffinwachs und von 85 bis 92 Gew.% nichtionischem oberflächenaktiven Mittel
50 besteht.

Revendications

- 55 1. Composition détergente en poudre séchée par vaporisation ne contenant substantiellement pas de phosphate inorganique et comprenant au moins 5 % en masse d'un ou de plusieurs agents tensioactifs anioniques, de 20 à 80 % en poids d'édificateur de détergence aluminosilicate cristallin ou amorphe et pas plus de 10 % en poids de silicate de métal alcalin, et de 0,1 à 6,0 % en masse d'une cire de paraffine qui n'est pas soluble dans l'eau et substantiellement non soluble dans les agents tensioactifs anioniques et non ioniques et a un point de fusion

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compris dans la gamme allant de 30 à 100°C et un angle de contact avec l'eau d'au moins 75°, **caractérisée en ce que** la poudre peut être obtenue à partir d'un procédé incluant l'étape consistant à sécher par vaporisation une pâte aqueuse des ingrédients détergentes, la cire de paraffine étant soit incorporée dans la pâte, soit vaporisée sur la poudre séchée par vaporisation, et **en ce que** la poudre ne contient substantiellement pas d'huile de paraffine.

2. Poudre selon la revendication 1, **caractérisée en ce que** la cire de paraffine à un angle de contact avec l'eau d'au moins 85°.

3. Poudre selon la revendication 1 ou la revendication 2, **caractérisée en ce que** la cire de paraffine a un point de fusion compris dans la gamme allant de 40 à 80°C.

4. Poudre selon l'une quelconque des revendications précédentes, **caractérisée en ce que** la quantité de cire de paraffine incorporée dans la poudre est comprise dans la gamme allant de 0,2 à 4,0 % en masse.

5. Poudre selon l'une quelconque des revendications précédentes, **caractérisées en ce qu'elle** a une densité en masse d'au moins 450 g/litre.

6. Poudre selon la revendication 5, **caractérisée en ce qu'elle** a une densité en masse d'au moins 600 g/litre.

7. Procédé de préparation d'une poudre détergente ne contenant substantiellement pas de phosphate inorganique et comprenant au moins 5 % en poids d'un ou de plusieurs tensioactifs anioniques, de 20 à 80 % en poids d'édificateur de détergence aluminosilicate cristallin ou amorphe et pas plus de 10 % en poids de silicate de métal alcalin et de 0,1 à 6 % en masse, sur la base de la poudre, d'une cire de paraffine qui n'est pas soluble dans l'eau et qui est substantiellement non soluble dans les tensioactifs anioniques et non ioniques et qui a un point de fusion qui est compris dans la gamme allant de 30 à 100°C et un angle de contact avec l'eau d'au moins 70°, ledit procédé incluant l'étape consistant à vaporiser une pâte aqueuse, **caractérisé en ce que** la cire de paraffine est soit incorporée dans la pâte, soit vaporisée sur la poudre séchée par vaporisation et **en ce que** la poudre ne contient substantiellement pas d'huile de paraffine.

8. Procédé selon la revendication 7, **caractérisé en ce que** la cire de paraffine est incorporée par l'intermédiaire de la pâte, dans une quantité allant de 0,2 à 4,0 % en masse, sur la base de la poudre.

9. Procédé selon la revendication 8, **caractérisé en ce que** la cire de paraffine est pré mélangée avec l'agent tensioactif non ionique avant d'être mélangée avec les autres ingrédients de la pâte.

10. Procédé selon la revendication 7, **caractérisé en ce que** la cire de paraffine sous forme liquéfiée est vaporisée sur la poudre séchée par vaporisation dans une quantité allant de 0,2 à 4,0 % en masse, sur la base de la poudre ainsi obtenue.

11. Procédé selon la revendication 10, **caractérisé en ce qu'il** comprend le fait d'appliquer sur la poudre une composition d'enrobage essentiellement composé de :

(i) de 2 à 50 % en masse de la cire de paraffine dispersée dans

(ii) de 50 à 98 % en masse de tensioactif anionique

les pourcentages étant sur la base de la composition d'enrobage.

12. Procédé selon la revendication 11, **caractérisé en ce que** la composition d'enrobage est essentiellement composée de 5 à 30 % en poids de cire de paraffine et de 70 à 95 % en poids de tensioactif non ionique.

13. Procédé selon la revendication 12, **caractérisé en ce que** la composition d'enrobage est essentiellement composée de 8 à 15 % en poids de cire de paraffine et de 85 à 92 % en masse de tensioactif non ionique.