

12

EUROPEAN PATENT APPLICATION

21 Application number: **89200587.7**

51 Int. Cl.⁴: **C11D 3/18 , C11D 11/02**

22 Date of filing: **09.03.89**

30 Priority: **21.03.88 GB 8806702**

43 Date of publication of application:
18.10.89 Bulletin 89/42

84 Designated Contracting States:
CH DE ES FR GB IT LI NL SE

71 Applicant: **UNILEVER NV**
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)

84 **CH DE ES FR IT LI NL SE**

Applicant: **UNILEVER PLC**
Unilever House Blackfriars P.O. Box 68
London EC4P 4BQ(GB)

84 **GB**

72 Inventor: **Appel, Peter Willem**
Prinses Julianalaan 79
NL-3062 DG Rotterdam(NL)
Inventor: **Kielman, Hendrik Simon**
Wegedoorn 4
NL-3142 KC Maassluis(NL)
Inventor: **Liem, Seeng Djiang**
Ghijseland 238
NL-3161 VR Rhoon(NL)
Inventor: **Waas, Marco**
St. Jacobstraat 139
NL-3011 DK Rotterdam(NL)

74 Representative: **Tan, Bian An, Ir. et al**
Unilever N.V. Patent Division P.O. Box 137
NL-3130 AC Vlaardingen(NL)

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

EP 0 337 523 A1

57 Dispensing of certain spray-dried detergent powders in automatic washing machines is substantially improved by the incorporation of 0.1-6.0% by weight of a hydrophobic additive having a contact angle to water of at least 75° and a melting point of 30-100° C. The additive may be included in the slurry from which the powder is prepared, or sprayed, preferably dispersed in nonionic surfactant, onto the powder. The preferred additive is paraffin wax.

DETERGENT POWDERS AND PROCESSES FOR PREPARING THEM

TECHNICAL FIELD

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
 5 powders containing no, or reduced levels of, phosphate builders and to powders of high bulk density.

BACKGROUND

10 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 powders to dispense less well in front-loading automatic
 15 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.

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

20 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 hydrophobic materials, either via the slurry or by subsequent spray-on.

Surprisingly, the incorporation of these materials via the slurry also improves powder structure and, when anionic surfactant is present, raises the bulk density.

25

DEFINITION OF THE INVENTION

In a first aspect, the invention comprises a spray-dried detergent powder comprising one or more
 30 anionic and/or nonionic surfactants and further comprising an additive which is a water-insoluble material substantially insoluble in anionic and nonionic surfactants, having a melting point within the range of from 30 to 100°C and having a contact angle to water of at least 75°, the additive being present in an amount of from 0.1 to 6.0% by weight and the powder giving a dispenser residue at least 10% by weight less than that of a comparative powder without the additive, as measured in a Philips (Trade Mark) AWB 126/7
 35 washing machine using 100 g powder and 5 litres of water at 20°C over a period of 1 minute.

In a second aspect, the invention provides a process for the preparation of a detergent powder comprising one or more anionic and/or nonionic surfactants and further comprising an additive which is a water-insoluble material substantially insoluble in anionic and nonionic surfactants, having a melting point within the range of from 30 to 100°C and having a contact angle to water of at least 75°, the process
 40 including the step of spray-drying an aqueous slurry, the additive being incorporated in the slurry or sprayed onto the powder in an amount of from 0.1 to 6% by weight based on the powder, and the powder thus obtained giving a dispenser residue at least 10% by weight less than that of a comparative powder without the additive, as measured in a Philips (Trade Mark) AWB 126/7 washing machine using 100 g powder and 5 litres of water at 20°C over a period of 1 minute.

45

PRIOR ART

Detergent compositions containing foam control systems that include hydrocarbon waxes are disclosed
 50 in EP 87 233A, EP 109 247A and EP 206 522A (Unilever); GB 1 492 938, GB 1 492 939, GB 2 040 982B, EP 8 829A, EP 8 830A and JP 56 034797A (Procter & Gamble); DE 3 436 194A, US 4 590 194 and EP 150 386A (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

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.

As essential ingredients, the detergent powder of the invention contains one or more anionic and/or nonionic surfactants, and a specified hydrophobic material that influences its dispensing behaviour in a particular manner.

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

The surfactant component

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.

The invention is of especial applicability 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 an additive such as 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 desirably 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.

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.

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.

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 0:1, 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 hydrophobic material

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 hydrophobic material. The hydrophobicity of the material is expressed in terms of its contact angle to water, which must be at least 75°, preferably at least 85°. Hydrocarbons are especially preferred.

The melting point of the material is also important: it appears that the additive 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 additive should lie within the range of from 30 to 100°C, preferably from 40 to 80°C. Preferred materials are paraffin waxes melting within the specified range, and preferably melting 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.

It is also essential that the hydrophobic material be substantially insoluble in the anionic and nonionic

surfactant system present in the composition: the microcrystalline waxes of GB 1 492 938 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.

5 The hydrophobic material 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 hydrophobic material

10

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 fluoreseers. More heat-sensitive ingredients can be postdosed to, or sprayed onto, the spray-dried base.

15

There are two methods by means of which the hydrophobic material 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 hydrophobic material with surfactant, especially nonionic surfactant, before admixture with other slurry ingredients.

20

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.

25

The second method by which the hydrophobic material 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 hydrophobic material should be sprayed on after addition of those ingredients so as to cover the whole powder.

30

If desired, the hydrophobic material may be melted and sprayed directly onto the powder. The hydrophobic material 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.

35

According to a preferred embodiment of the invention, however, the hydrophobic material is premixed with nonionic surfactant to form a coating composition which may then be sprayed onto the detergent powder. Lower levels of the hydrophobic material, 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 hydrophobic material and 50-98% by weight of nonionic surfactant. In the preferred embodiment of the invention in which the hydrophobic material is paraffin wax, 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.

40

The coating composition should be substantially free of other ingredients that might interfere with the beneficial effect of the hydrophobic material on dispensing.

45

Dispensing behaviour

50

It is an essential feature of the detergent powder of the invention that the incorporation as specified above of a hydrophobic material such as 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 hydrophobic material 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.

55

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 additive, 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 550 g/litre or more.

Optional components

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, sodium silicate, bleaches, fluorescers, polymers, lather control agents, enzymes and perfumes.

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.

The powder will also contain one or more detergency builders. The invention is of especial applicability, as previously mentioned, to low- or zero-phosphate powders containing crystalline (zeolite) or amorphous aluminosilicate. Zeolite may suitably be present in an amount of from 20 to 80% by weight. 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.

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 is of especial applicability to powders containing less than 10% by weight, more especially less than 5% by weight, of sodium silicate.

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.

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

EXAMPLES

Examples 1 to 4

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	0, 0.25, 0.5, 1.0, 2.0
or paraffin oil	1.0
Total	51-53 parts

*Sokalan (Trade Mark) CP5 ex BASF.

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

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	

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.

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

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

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

Example 5

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.

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

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

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

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.

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.

	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

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

5

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:

10

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

25

Dispensing and powder properties were as follows:

30

35

	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		

40

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 comprising one or more anionic and/or nonionic surfactants and further comprising an additive which is a water-insoluble material substantially insoluble in anionic and nonionic surfactants, having a melting point within the range of from 30 to 100 °C and having a contact angle to water of at least 75°, characterised in that the additive is present in an amount of from 0.1 to 6.0% by weight and in that the powder gives a dispenser residue at least 10% by weight less than that of a comparative powder without the additive, as measured in a Philips (Trade Mark) AWB 126/7 washing machine using 100 g powder and 5 litres of water at 20 °C flowing in over a period of 1 minute.

2. A powder as claimed in claim 1, characterised in that it gives a dispenser residue in the defined test at least 20% by weight less than that of the comparative powder without the additive.

3. A powder as claimed in claim 1 or claim 2, characterised in that the comparative powder without the additive gives a dispenser residue of at least 10% by weight in the defined test.

4. A powder as claimed in claim 3, characterised in that the comparative powder without the additive gives a dispenser residue of at least 20% by weight in the defined test.

5. A powder as claimed in any preceding claim, characterised in that the additive has a contact angle to water of at least 85°.

5 6. A powder as claimed in any preceding claim, characterised in that the additive has a melting point within the range of from 40 to 80°C.

7. A powder as claimed in any preceding claim, characterised in that the additive is a hydrocarbon.

8. A powder as claimed in claim 7, characterised in that the additive is a paraffin wax.

9. A powder as claimed in claim 5 or claim 6, characterised in that the amount of additive incorporated
10 in the powder is within the range of from 0.2 to 4.0% by weight.

10. A powder as claimed in any preceding claim, characterised in that it is substantially free from inorganic phosphate.

11. A powder as claimed in any preceding claim, characterised in that it contains at least 5% by weight of one or more anionic surfactants.

12. A powder as claimed in any preceding claim, characterised in that it comprises from 20 to 80% by weight of crystalline or amorphous aluminosilicate detergency builder.

13. A powder as claimed in any preceding claim, characterised in that it contains no more than 10% by weight of alkali metal silicate.

14. A powder as claimed in any preceding claim, characterised in that it has a bulk density of at least
20 450 g/litre.

15. A powder as claimed in claim 14, characterised in that it has a bulk density of at least 600 g/litre.

16. A process for the preparation of a detergent powder comprising one or more anionic and/or nonionic surfactants and further comprising an additive which is a water-insoluble material substantially insoluble in anionic and nonionic surfactants, having a melting point within the range of from 30 to 100°C and having a
25 contact angle to water of at least 75°, the process including the step of spray-drying an aqueous slurry, characterised in that the additive is incorporated in the slurry or sprayed onto the powder in an amount of from 0.1 to 6% by weight based on the powder, and in that the powder thus obtained gives a dispenser residue at least 10% by weight less than that of a comparative powder without the additive, as measured in a Philips (Trade Mark) AWB 126/7 washing machine using 100 g powder, and 5 litres of water at 20°C
30 flowing in over a period of 1 minute.

17. A process as claimed in claim 16, characterised in that the additive is incorporated via the slurry, in an amount of from 0.2 to 4.0% by weight based on the powder.

18. A process as claimed in claim 17, characterised in that the additive is premixed with nonionic surfactant before admixture with other slurry ingredients.

19. A process as claimed in claim 18, characterised in that the additive in liquefied form is sprayed onto the spray-dried powder, in an amount of from 0.2 to 4.0% by weight based on the powder thus obtained.

20. A process as claimed in claim 19, characterised in that it comprises spraying onto the powder a coating composition consisting essentially of:

(i) from 2 to 0% by weight of the additive dispersed in

40 (ii) from 50 to 98% by weight of nonionic surfactant, the percentages being based on the coating composition.

21. A process as claimed in claim 20, characterised in that the additive is paraffin wax, and the coating composition consists essentially of from 5 to 30% by weight of paraffin wax and from 70 to 85% by weight of nonionic surfactant.

22. A powder as claimed in claim 21, characterised in that the coating composition consists essentially
45 of from 8 to 15% by weight of paraffin wax and from 85 to 92% by weight of nonionic surfactant.

50

55



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 A	US-A-2 423 450 (R. FRANKLIN et al.) * whole document *	1,9,16, 17,19 7,10,11	C 11 D 3/18 C 11 D 11/02
Y A	EP-A-0 045 208 (UNILEVER) * page 4, lines 1-5,33-36; page 5, lines 12-22; page 7, lines 14-19; claims 1-4,6 *	1,9,16, 17,19 6,8,10- 13	
A,D	FR-A-2 257 680 (PROCTER & GAMBLE) * claims 1-5,7-10,24,25 *; & GB - A - 1492938 (PROCTER & GAMBLE)	1,6,8,9	
A,D	EP-A-0 206 522 (UNILEVER) * abstract, claims 1,4,6,10,16-19,22,23 *	1,6,8, 11,12	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			C 11 D
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 16-06-1989	Examiner PELLI-WABLAT B
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			