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54 **Process for preparing detergent powders having improved dispensing properties.**

57 The dispensing of a detergent powder in an automatic washing machine is improved by spraying on an intimate mixture of a fatty acid and a nonionic surfactant.

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PROCESS FOR PREPARING DETERGENT POWDERS HAVING IMPROVED DISPENSING PROPERTIES

TECHNICAL FIELD

The present invention relates to detergent compositions having improved dispensing properties in automatic front-loading washing machines. The invention is of especial applicability to detergent powders of high bulk density.

BACKGROUND

Recently there has been a trend within the detergents industry towards powders having higher bulk densities, for example, 450 g/l and above, than has been customary in the past. A problem that has been encountered with these denser powders is a tendency to dispense less well in front-loading automatic washing machines; a higher proportion of the powder dosed into the machine is left behind in the dispenser, leading to powder wastage and clogging. This problem is especially marked at low wash temperatures.

We have now found that the dispensing properties of detergent powders may be substantially improved by spraying on to the powders, at some stage in their manufacture, an intimate mixture of a fatty acid and a nonionic surfactant.

PRIOR ART

The European patent application 142,910 (Procter & Gamble) discloses detergent powders prepared by spray-drying an aqueous slurry containing surfactants, builders, salts and other conventional components, then post-dosing silicone suds suppressor, enzyme and bleach components; additional nonionic surfactant and fatty acid (1% or 1.5%) are sprayed on in some Examples. The fatty acid is included in order to improve suds suppression.

DEFINITION OF THE INVENTION

The present invention provides a process for the preparation of a detergent powder, which includes the steps of preparing an intermediate powder, and spraying on to the intermediate powder an intimate mixture of a C₈-₂₂ fatty acid and a liquid or liquefiable nonionic surfactant, the fatty acid being employed in an amount of at least 0.10% by weight, based on the final detergent powder, and the final detergent powder having a dynamic flow rate (as hereinafter defined) of at least 90 ml/s.

The invention also provides a detergent powder prepared by the process defined in the previous paragraph.

Another aspect of the invention is the use of the intimate mixture of a fatty acid and a nonionic surfactant for improving the dispensing properties of a detergent powder.

DETAILED DESCRIPTION OF THE INVENTION

In the process of the invention, the dispensing properties of a detergent powder are improved by spraying on an intimate mixture of a fatty acid and a liquid or liquefiable nonionic surfactant at some suitable stage during the manufacture of the powder. This spray-on process may be the final stage in the manufacture of the powder; or it may precede the addition of other components.

High-performance detergent powders are conventionally prepared by spray-drying an aqueous slurry of heat-insensitive and mutually compatible ingredients to form a spray-dried granular product, normally referred to as the base powder. Other desired ingredients that are not to be incorporated via the slurry, because for example of heat sensitivity or incompatibility with other slurry ingredients, are then dry-mixed (solids) or sprayed on (liquids).

In a process of this type, the intimate mixture of fatty acid and nonionic surfactant employed according to the present invention may conveniently be sprayed either on to the base powder, prior to the dry-dosing and/or spraying of other components; or it may be sprayed on to the final product; or some intermediate

procedure may be used.

Thus a first embodiment of the invention comprises the steps of:

- (i) preparing a base powder by spray-drying an aqueous slurry,
- (ii) spraying the fatty acid/nonionic surfactant mixture on to the base powder obtained in step (i), and
- 5 (iii) post-dosing one or more other ingredients to the base powder obtained in step (ii) to give the final detergent powder.

A second embodiment of the invention comprises the steps of:

- (i) preparing a base powder by spray-drying an aqueous slurry,
- (ii) post-dosing one or more other ingredients to the base powder to form an intermediate powder,
- 10 and
- (iii) spraying the fatty acid/nonionic surfactant mixture on to the intermediate powder obtained in step (ii) to give the final detergent powder.

In each of these embodiments, the base powder prepared in step (i) may, if desired, be modified by a subsequent treatment, for example to increase its bulk density or change its character, before steps (ii) and

15 (iii) are carried out. Of course the process of the invention is also applicable to powders prepared by non-spray-drying processes.

Where the powder would otherwise display very poor dispensing characteristics in the automatic front-loading washing machine, the process of the invention has been found to result in a substantial improvement in these characteristics. Thus the process of the invention is especially applicable to those powders

20 which, if prepared by an identical process minus the fatty acid, would give residues of 25 g or more, more especially 50 g or more, in the test described below. For the purposes of the present invention, dispensing is assessed using a dispenser drawer as fitted to a Hoover Matchbox (Trade Mark) 3263H washing machine. A 200 g dose of powder is placed in the main

25 compartment of the drawer, and water at 10 °C is allowed to flow through at a rate of 2 litre/min. for 2 minutes. At the end of the 2-minute period, the flow is stopped, excess water is poured off, and the residual mass of wet powder weighed. This test represents very stringent conditions of low water temperature and a slow water fill, deliberately chosen to be more severe than those encountered in normal usage: a wet residue of 50 g or less under these conditions may be regarded as acceptable, and a wet residue of 25 g or

30 less as good. A detergent powder prepared in accordance with the invention preferably gives a dispenser residue at least 20 g less, more preferably at least 50 g less, than that of a comparative powder prepared by an otherwise identical process without the sprayed-on fatty acid. In the manufacture of the comparative powder, the nonionic surfactant alone should be sprayed on, without the fatty acid.

35 The amount of fatty acid sprayed on is at least 0.1% by weight, based on the final powder. The maximum amount of fatty acid that may be sprayed on is limited only by its tendency to have a detrimental effect on the flow properties of the powder. For the purposes of the present invention, powder flow is defined in terms of the dynamic flow rate, in ml/s, measured by means of the following procedure. The apparatus used consists of a cylindrical glass tube having an internal diameter of 35 mm and a length of

40 600 mm. The tube is securely clamped in a position such that its longitudinal axis is vertical. Its lower end is terminated by means of a smooth cone of polyvinyl chloride having an internal angle of 15 and a lower outlet orifice of diameter 225 mm. A first beam sensor is positioned 150 mm above the outlet, and a second beam sensor is positioned 250 mm above the first sensor.

To determine the dynamic flow rate of a powder sample, the outlet orifice is temporarily closed, for

45 example, by covering with a piece of card, and powder is poured through a funnel into the top of the cylinder until the powder level is about 10 cm higher than the upper sensor; a spacer between the funnel and the tube ensures that filling is uniform. The outlet is then opened and the time t (seconds) taken for the powder level to fall from the upper sensor to the lower sensor is measured electronically. The measurement is normally repeated two or three times and an average value taken. If V is the volume (ml) of the tube

50 between the upper and lower sensors, the dynamic flow rate DFR (ml/s) is given by the following equation:

$$DFR = V / t$$

The averaging and calculation are carried out electronically and a direct read-out of the DFR value obtained.

Preferably, the final powder, after treatment in accordance with the invention, has a dynamic flow rate of at least 90 ml/s, more preferably at least 100 ml/s. The process of the invention is thus especially suitable

55 for treating powders that, in the absence of fatty acid spray on, have especially good flow properties, for example a dynamic flow rate of at least 110 ml/s, preferably at least 120 ml/s.

The amount of fatty acid spray-on that can be tolerated without reducing the dynamic flow rate to an unacceptable level is generally greater, the larger the particle size of the powder. For powders having an

average particle size of 1 mm or less, the amount of fatty acid sprayed on preferably does not exceed 1% by weight based on the final powder. A range of 0.1 to 1% by weight is preferred, a range of 0.2 to 0.8% by weight is more preferred, and a range of 0.3 to 0.5% by weight is especially suitable. For powders having an average particle size greater than 1 mm, higher fatty acid levels can be tolerated.

5 Good results have been obtained if the fatty acid is wholly or predominantly composed of C₁₅₋₂₂ saturated fatty acids. Suitable fatty acids are those derived from hardened oils and fats; for example, tallow, palm oil, rapeseed oil and marine oils hardened to an iodine value of less than 20 and preferably less than 5.

These relatively long-chain fatty acids containing only low levels of unsaturated material are also more stable, and give the additional benefit of contributing to lather control in the product.

10 It is an essential feature of the process of the invention that the fatty acid be mixed intimately with a liquid or liquefiable nonionic surfactant before being sprayed on to the powder. The amount of nonionic surfactant employed is preferably within the range of from 1 to 10% by weight, more preferably from 2 to 5% by weight, based on the final detergent powder. Additional nonionic surfactant may also be included, independently of the spray-on process, in the final product.

Any suitable nonionic surfactant that is liquid or readily liquefiable at temperatures up to about 65° C may be used. A preferred type of nonionic surfactant for this purpose is a C₁₂₋₁₅ aliphatic alcohol ethoxylated with 2 to 10 moles of ethylene oxide per mole of alcohol.

15 It has been found that intimate admixture of the fatty acid and the nonionic surfactant prior to spray-on is essential in order to obtain the full benefit of the invention. Sequential separate addition of the two materials, in either order, is substantially less effective.

The weight ratio of nonionic surfactant to fatty acid is preferably at least 1:1, more preferably at least 2:1, to aid spreading of the fatty acid over the powder particles.

25 The spray-on process is preferably carried out using apparatus that will ensure the most thorough coverage possible of the particle surfaces by the sprayed-on mixture. A rotating drum, for example, is suitable. The more efficient the spray-on process, the lower the level of fatty acid that needs to be used. The mixture is preferably sprayed on at a temperature between its melting point and 95° C. Conveniently, the mixture is sprayed, at a temperature substantially above its melting point, on to cool powder: 60° C has been found to be a suitable temperature.

30 The invention is of especial applicability to the preparation of powders of high bulk density, for example, at least 450 g/l. The process of the invention is especially valuable for treating very high bulk density (≥700 g/l) powders which combine excellent flow with poor dispensing properties, since high levels of fatty acid can be tolerated without lowering the dynamic flow rate to an unacceptable level.

35 The invention is also of especial applicability to powders containing silicone oil as an anti-foam ingredient. Silicone oil is generally incorporated in detergent powders by post-spraying or post-dosing procedures; for example, it may be adsorbed on a solid carrier and the resulting adjunct then dry-mixed with the spray-dried base powder. Examples of suitable solid carriers for silicone oil include porous carbonate-based materials as disclosed in EP 266 863A (Unilever), for example light soda ash and crystal-growth modified Burkeite; zeolite; and microporous sodium perborate monohydrate, as disclosed in our co-
40 pending European patent application No. 89 30505.7. In this embodiment of the invention, the fatty acid/nonionic surfactant mixture may be sprayed on either before or, preferably, after the addition of the anti-foam adjunct.

Detergent powders prepared by the process of the invention may contain any of the ingredients conventionally present in compositions intended for the washing of fabrics in automatic front-loading
45 washing machines. Such ingredients include a surfactant system; a phosphate or non-phosphate builder system; a bleach system; sodium silicate; other inorganic salts; fluoresters; anti-redeposition agents; anti-foam ingredients; enzymes; other performance ingredients; pigments; and perfumes. This list is not intended to be exhaustive but merely to show the very general applicability of the process of the invention.

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

Example 1

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Detergent powders were prepared to the following formulations by conventional spray-drying and post-dosing processes.

		<u>1</u>	<u>A</u>
	<u>Base powder</u>		
	Linear alkylbenzene sulphonate	6.0	6.0
5	Nonionic surfactant (7EO) ¹	4.0	4.0
	Sodium tripolyphosphate	26.0	26.0
	Sodium alkaline silicate (2.0r)	8.0	8.0
10	Sodium sulphate	15.7	15.7
	Minor ingredients, moisture	11.0	11.0
	<u>Sprayed on</u>		
15	Fatty acid ²	) 0.2	0
	Nonionic surfactant (3EO) ³	) 3.0	3.0
	Perfume	0.1	0.1
20			
	<u>Post-dosed</u>		
25	Sodium perborate tetrahydrate	15.0	15.0
	Sodium carbonate (granular)	10.0	10.2
	Lather control granule	1.0	1.0
30		-----	
		100.0	100.0
	1 Synperonic (Trade Mark) A7 ex ICI: C ₁₂ -C ₁₅		
35	alcohol 7EO ethoxylate.		
	2 Pristerene (Trade Mark) 4916: fully hardened tallow		
	fatty acid.		
40	3 Synperonic (Trade Mark) A3 ex ICI: C ₁₂ -C ₁₅ alcohol		
	3EO ethoxylate.		

45 The spray-dried base powder had a mean particle size of 560 microns and a moisture content of about 10%. It was cooled to a temperature of about 30° C by airlifting. Two kg batches were taken for further processing in a small Benford concrete mixer, one (Example 1) being in accordance with the invention and the other (Comparative Example A) being a control. Prior to spray-on (Example 1 only) the fatty acid was dispersed in the nonionic surfactant (3EO) at a temperature of about 60° C, and the spraying-on was carried out in a rotating concrete mixer. Perfume was then sprayed on, and finally the sodium perborate

50 tetrahydrate, granular sodium carbonate and lather control granules were added to give a finished detergent powder. A similar procedure was followed for Comparative Example A, except that the nonionic surfactant sprayed on contained no fatty acid. Dispenser residues were measured by the test described previously, four replicas being carried out for each powder. Average results were as follows:

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Example 1	17 g
Comparative Example A	114 g

Example 2

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The procedure of Example 1 was repeated using a slightly different base powder containing 6 parts of anionic surfactant (sodium linear alkylbenzene sulphonate) and 4.5 parts of nonionic surfactant 7EO, the parts here being equivalent to percentages of a notional fully formulated product. 0.3 parts of fully hardened
 10 tallow fatty acid dispersed in 3.5 parts of nonionic surfactant 3EO were sprayed onto the base, which then gave a dispenser residue of zero, and had a dynamic flow rate of 106 ml/s.

Example 3

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These examples show that pre-mixture of the fatty acid and the nonionic surfactant is necessary in order to obtain the dispensing benefit of the invention, and also shows the effect of fatty acid on powder flow.

20 In a procedure similar to that of Example 1, a detergent base powder was prepared by a conventional spray-drying process. This contained 7 parts of anionic surfactant and 1 part of nonionic surfactant (7EO), the parts here being equivalent to percentages of a notional final product, but was otherwise similar to that of Example 1.

The base powder was sprayed with 2 parts of nonionic surfactant (3EO) and 1 part of fully hardened
 25 tallow fatty acid in various combinations as follows :

30

Example	Spray-on
B	nonionic only
C	fatty acid, followed by nonionic
D	nonionic, followed by fatty acid
3	mixture of fatty acid and nonionic

35

The dispenser residues and dynamic flow rates of the powders were as follows :

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Example	Dispenser residue (g)	Dynamic flow rate (ml/s)
Base	12	110
B	29	110
C	69	110
D	48	110
3	0	93

45

Thus spray-on of nonionic surfactant alone, and sequential spray-on of nonionic surfactant and fatty acid in either order, all caused a deterioration in the dispensing properties of the base powder. Spray-on of
 50 an intimate mixture of fatty acid and nonionic surfactant eliminated dispenser residues, but at the expense of some deterioration of the flow properties of the powder.

Example 4

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This Example illustrates an embodiment of the invention in which a fatty acid/nonionic surfactant mixture is sprayed on to a powder after post-dosing of solid ingredients.

52 parts of a base powder similar to that of Example 1 but containing less sodium sulphate was prepared by spray-drying; 3 parts of nonionic surfactant 3EO were sprayed on; 45 parts of solid ingredients (sodium perborate, sodium sulphate, sodium carbonate and lather control granules (containing silicone oil on a light soda ash carrier) were post-dosed; and then an intimate mixture of 1 part of fully hardened tallow, fatty acid and 1 part of nonionic surfactant was sprayed on to the resulting powder.

The properties of the base, the product prior to spray-on, and the product after spray-on, were as follows :

	Base	Before	After
Bulk density (g/l)	533	717	650
Dynamic flow rate (ml/s)	123	127	100
Dispenser residue (g)	0	98	1

It will be seen that the dispenser residue was virtually eliminated, but at the cost of some loss of flow.

Claims

1. A process for the preparation of a detergent powder, which includes the steps of preparing an intermediate powder, and spraying on to the intermediate powder an intimate mixture of a C_{8-22} fatty acid and a liquid or liquefiable nonionic surfactant, the fatty acid being employed in an amount of at least 0.1% by weight, based on the final detergent powder, the final detergent powder having a dynamic flow rate (as hereinbefore defined) of at least 90 ml/s.
2. A process as claimed in Claim 1, which comprises the steps of:
 - (i) preparing a base powder by spray-drying an aqueous slurry,
 - (ii) post-dosing one or more other ingredients to the base powder to form an intermediate powder, and
 - (iii) spraying the fatty acid/nonionic surfactant mixture on to the intermediate powder obtained in step (ii) to give the final detergent powder.
3. A process as claimed in Claim 1, which comprises the steps of:
 - (i) preparing a base powder by spray-drying an aqueous slurry,
 - (ii) spraying the fatty acid/nonionic surfactant mixture on to the base powder obtained in step (i), and
 - (iii) post-dosing one or more other ingredients to the base powder obtained in step (ii) to give the final detergent powder.
4. A process as claimed in any preceding Claim, wherein the final detergent powder gives a dispenser residue, in a test hereinbefore defined, at least 20 g less than that of a comparative powder prepared by an otherwise identical process without the sprayed-on fatty acid.
5. A process as claimed in Claim 4, wherein the comparative powder gives a dispenser residue greater than 25 g in the test hereinbefore defined.
6. A process as claimed in any preceding Claim, wherein the amount of fatty acid sprayed on is within the range of from 0.2 to 0.5% by weight, based on the final detergent powder.
7. A process as claimed in any preceding Claim, wherein the fatty acid is wholly or predominantly composed of C_{16-22} saturated fatty acids.
8. A process as claimed in any preceding Claim, wherein the weight ratio of nonionic surfactant to fatty acid in the intimate mixture sprayed on is at least 1:1.
9. A process as claimed in any preceding Claim, wherein the final detergent powder has a bulk density of at least 450 g/litre.
10. Use of an intimate mixture of a C_{8-22} fatty acid and a liquid or liquefiable nonionic surfactant for improving the dispensing properties of a detergent powder.