(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 087 233**
A1

(12) # EUROPEAN PATENT APPLICATION

(21) Application number: 83300540.8

(22) Date of filing: 03.02.83

(51) Int. Cl.³: C 11 D 3/32
C 11 D 3/26, C 11 D 3/00

(30) Priority: 04.02.82 GB 8203233

(43) Date of publication of application:
31.08.83 Bulletin 83/35

(84) Designated Contracting States:
AT BE CH DE FR GB IT LI NL SE

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(84) Designated Contracting States:
GB

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(84) Designated Contracting States:
BE CH DE FR IT LI NL SE AT

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(54) **Process for producing low-sudsing detergent compositions.**

(57) A process for making low-sudsing detergent powders is disclosed. The particular feature of the process is the formation of an intimate mixture of an oily or waxy substance and a defined nitrogen compound, an α,ω-dialkylamide alkane for example, and the subsequent spraying of the mixture onto either a carrier, or onto spray-dried detergent powder.

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PROCESS FOR PRODUCING LOW-SUDSING DETERGENT COMPOSITIONS

This invention relates to a process for producing low-sudsing detergent compositions, and to the compositions produced.

Low-sudsing detergent compositions are increasing in popularity as the number of front-loading, drum-type washing machines increases, and are now widespread. They are generally in spray-dried, particulate form, although liquids are now being introduced, and are formulated with a high sudsing detergent system together with a suds-suppressant. The high sudsing detergent system is usually either an anionic detergent-active compound alone, or a combination of an anionic and a nonionic detergent-active compound. The suds-suppressant component can be selected from a wide variety of types, examples being alkyl phosphoric acid esters and their salts, hydrocarbons such as petroleum jelly and waxes, silicone oils and hydrophobed silicas, and mixtures thereof.

In British Patent Specification No 1,324,289 and in German DOLS 2,043,087 low-sudsing compositions containing various complex nitrogen compounds as suds control agents are described. We have now discovered that if these compounds are intimately admixed with an oily or waxy substance and incorporated into what would otherwise be a high-sudsing spray-dried detergent composition by spraying, then excellent low-sudsing compositions are produced.

Accordingly the present invention provides a process for the manufacture of a low-sudsing detergent composition which comprises forming an essentially spray-dried detergent composition and adding a suds-suppressant to it by spraying, the suds-suppressant comprising an intimate mixture of an oily or waxy substance and a nitrogen compound of the general formula

(i) $R^1-N(R^2)-C(=O)-C(=O)-N(R^4)-R^3$

(ii) $R^1-N(R^2)-C(=O)-R-C(=O)-N(R^4)-R^3$

(iii) $R^1-O-C(=O)-NH-R-NH-C(=O)-O-R^2$

or

(iv) $R^1-C(=O)-N(R^2)-R-N(R^4)-C(=O)-R^3$

where R^1 and R^3 represent aliphatic hydrocarbon residues containing 14-22 carbon atoms, R^2 and R^4 represent hydrogen atoms or aliphatic hydrocarbon residues containing 1-22 carbon atoms, and R represents an aliphatic residue containing 1-9 carbon atoms.

Preferred compounds are α,ω-dialkylamide alkanes, that is to say compounds of the general formula (iv) above, and of these α,ω-distearylamide methane or ethane (commonly known as methylene and ethylene distearamides) that is compounds of the general formula:

$$C_{17}H_{35}-\underset{\underset{O}{\|}}{C}-NH-(CH_2)_n-NH-\underset{\underset{O}{\|}}{C}-C_{17}H_{35}$$

where n is 1 or 2, are most preferred.

Examples of oily substances are mineral, vegetable or animal oils, colourless mineral oils being preferred. Either light or heavy mineral oil or mixtures thereof may be employed but, of course, any substance used must be of low volatility at normal fabric-washing temperatures. Other oils which are suitable are sesame oil, cottonseed oil, corn oil, sweet almond oil, olive oil, wheat germ oil, rice bran oil or peanut oil, or animal oils such as lanolin, neat's foot oil, bone oil, sperm oil or cod liver oil.

Preferred waxy materials are of mineral origin, especially those derived from petroleum, including microcrystalline and oxidised microcrystalline petroleum waxes and paraffin waxes. A preferred material is petroleum jelly, (often sold under the trade mark "Vaseline" by Cheseborough-Ponds Limited), desirably one having a drop-melting point of from 20°-120°C, preferably 45-65°C. The term 'drop-melting point' as used herein is used in the sense defined in ASTM designation D127-63. Synthetic waxes, or Montan waxes, or natural waxes such as beeswax, candelilla and carnauba waxes may also be used if desired.

Any of these waxes described may be used alone or in admixture with other waxes.

The suds-suppressant combination may be added to the essentially high sudsing composition in an amount such that it forms from 0.1 to 5%, preferably 0.5 to 3.5% by weight of the final composition. The relative proportions of the nitrogen compound and the oily substance in the suds-suppressant combination are generally from 2.5:97.5 to 9:1, preferably 5:95 to 1:3, the ratios being expressed by weight.

The suds-suppressant combination may be mixed with a dispersing agent such as a nonionic surfactant. Typical nonionic surfactants may for example be either ethoxylated alcohols or sorbitan esters. The relative proportions of dispersing agent to the suds-suppressant combination are generally from 0.2:99.8 to 3:1.

The suds-suppressant combination may also be mixed with other antifoam ingredients such as fatty acids and their soaps, alkyl phosphoric acids and their salts with metal ions, and hydrophobed silica. The relative proportion of other ingredients to the nitrogen compounds (i)-(iv) is from 1:9 to 9:1.

The compositions prepared by the process of this invention will contain either an anionic, a nonionic, a zwitterionic or a cationic surfactant or a mixture of these. Other components which will normally be present include detergency builders, fabric conditioning agents, bleaching agents, bleach activators, corrosion inhibitors, anti-redeposition agents, fluorescers, stabilisers and a substantial proportion of moisture.

Typical anionic surfactants, which may be present in amounts of from about 2 to 35% by weight of the finished composition, are sodium alkylbenzene sulphonates, preferably the C_{10}-C_{14} alkyl compounds, sodium primary and secondary alkyl sulphates, preferably the C_{10}-C_{22} alkyl sulphates, sodium olefine sulphonates, preferably the C_{10}-C_{18} sulphonates, sodium alkane sulphonates and sodium alcohol ether sulphates. Soaps of fatty acids may also be present, preferably the sodium and potassium salts of C_{10}-C_{22} fatty acids, both saturated and unsaturated. Where soap is the sole anionic surfactant it may be present in an amount up to about 65% by weight of the finished composition, down to about ½% by weight when other anionic surfactants are present. Typical soaps which can be used are those formed from coconut oil, tallow and natural oils containing high proportions of oleic acid such as sunflower oil.

The compositions produced by the process of the invention can also contain nonionic surfactants, preferably ethoxylated primary and secondary alcohols of from 8 to 25 carbon atoms containing from 3 to 25 moles of ethylene oxide per mole of alcohol. These materials may be present in an amount of from 1 to 15% by weight, based on the weight of the finished composition.

Typical detergency builders which can be used are the water-soluble phosphates, carbonates, percarbonates and aluminosilicates, particularly the sodium and potassium salts of these compounds. Organic builders may also be used, examples being sodium carboxymethyloxysuccinate, sodium citrate, sodium polyacrylates and sodium nitrilotriacetate. Any of these compounds, or any other builder compound, in any suitable mixture, may be used in amounts of from 5 to 50% by weight of the finished composition.

It is emphasised that the components of the suds-suppressant must be in intimate mixture prior to spraying on to the essentially high sudsing spray-dried detergent composition, since we have discovered that if the two components are added separately, inferior suds control is produced.

The normal method for making powdered detergent compositions is to combine the spray-dried base powder with components such as sodium perborate, sodium sulphate, enzyme compositions and perfume in a dry-dosing step. This step may be performed in accordance with the invention either prior to, simultaneously with, or after the spraying of the essentially high sudsing spray-dried detergent composition with the intimate mixture of the nitrogen compound and the oily substance.

Another approach to incorporation of the intimate mixture is to spray it onto a carrier material which is then mixed with a spray dried detergent composition. Examples of such carrier materials include sodium tripolyphosphate and zeolite. Other more preferred materials include water soluble porous organic and inorganic materials suh as sodium perborate monohydrate starch, and mixtures thereof. Other carrier materials could be prepared by spray drying a slurried mixture of inorganic salts in for example the manner taught by European patent specification EP 22 998, British applications 2 005 715, 1 595 769, 1 595 770, 2 002 808 or US patent 3 984 527.

The invention will be further illustrated in the following examples.

Example 1

In a model experiment a spray-dried detergent powder suitable for fabric washing having the formulation shown below was dissolved in demineralised water to form a solution containing 10g/l of the formulation.

	Parts by Weight
Sodium dodecylbenzene sulphonate	7.5
C_{12}-C_{15} primary alcohol ethoxylate (7EO)	2.5
Sodium tripolyphosphate	33.0
Sodium silicate (alkaline)	8.0
Sodium sulphate	11.3
Sodium carboxymethylcellulose	1.0
Sodium perborate	18.8
Water and minor components	12.0

In two separate experiments 0.04 gms/l ethylene bis stearamide and 0.16 g/l mineral oil were dispersed in the solution. Dispersion was achieved either by hand-shaking for 60 seconds or ultrasonically. In the first experiment the two components were added to the solution separately, whereas in the second experiment they were first intimately mixed and then dispersed.

The foam generated by each of these two solutions, as compared with a control solution not containing anti-foam was then measured using a Ross-Miles dynamic foam meter at 300 ml/min flow rate. The results are shown in Table 1, from which it can be seen that it is only in the case where the ethylene distearamide is intimately mixed with mineral oil that effective foam control is obtained.

Table 1

Temperature (°C)	Foam Height (cms) Control (No antifoam)	Antifoam added separately	Antifoam intimately mixed
20	0	0	0
30	18	8	5
40	45	37	12
50	75	63	15
60	80	80	22
70	80	80	28
80	80	80	28
90	80	80	11
100	80	80	N.D.

Example 2

The following two detergent powders were produced by a process involving slurry making and counter-current spray-drying steps.

	Parts by Weight A	B
Sodium dodecylbenzene sulphonate	7.5	7.5
Tallow alcohol 18EO	2.5	2.5
Sodium tripolyphosphate	33.0	33.0
Sodium sulphate	11.3	11.3
Sodium silicate (alkaline)	8.0	8.0
Sodium carboxymethylcellulose	1.0	1.0
Water and minor components	12.0	12.0
Ethylene distearamide/wax mixture (50:50)	2.0	-

Two parts of the ethylene bis stearamide/wax mixture were sprayed onto spray-dried powder B, and then both powders were dry-dosed with 18.8 parts of sodium perborate.

The foam produced by each formulation was then assessed as described in Example 1. The results are shown

in Table 2.

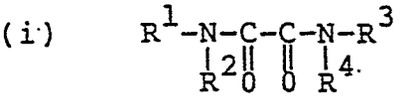
Table 2

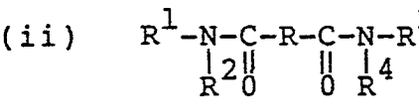
	Foam Heights (cms)	
	30 mins	end of wash
Powder A:	88°C overfoam	90°C overfoam
Powder B:	2.0	2.5

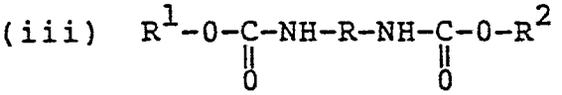
The deactivation of the antifoam processed by the through-slurry route is evident, as is the good retention of foam control of the powder sprayed with the intimate mixture of ethylene bis stearamide and wax.

CLAIMS

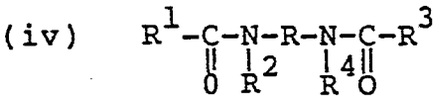
1. A process for the manufacture of a low-sudsing detergent composition which comprises forming an essentially spray-dried detergent composition and adding a suds-suppressant to it by spraying, the suds-suppressant comprising an intimate mixture of an oily or waxy substance and a nitrogen compound of the general formula

(i) 

(ii) 

(iii) 

or

(iv) 

where R^1 and R^3 represent aliphatic hydrocarbon residues containing 14-22 carbon atoms, R^2 and R^4 represent hydrogen atoms or aliphatic hydrocarbon residues containing 1-22 carbon atoms, and R represents an aliphatic residue containing 1-9 carbon atoms.

2. A process according to claim 1 wherein the compound is an α,ω-dialkylamide alkane of formula (iv).

3. A process according to claim 2 wherein the α,ω-dialkylamide alkane is α,ω-distearylamide methane or ethane.

4. A process according to claim any one of the preceding claims wherein the oily or waxy substance comprises a mineral oil, a petroleum jelly, a wax or a mixture thereof.

5. A process according to any one of the preceding claims wherein the suds-suppressant is sprayed onto the spray-dried powder in an amount such that it forms from 0.5 to 3.5% by weight of the composition.

6. A process according to any one of the preceding claims wherein the relative proportions by weight of the nitrogen compound and the oily substance in the suds-suppressant are from 2.5 to 97.5 to 9:1.

7. A process according to claim wherein the relative proportions by weight are 5:95 to 1:3.

8. A process according to any one of the preceding claims wherein the suds-suppressant comprises a dispersing agent in the form of a nonionic surfactant.

9. A process according to claim 8 wherein the relative proportions, by weight, of dispersing agent to suds-suppressant are from 0.2:99.8 to 3:1.

10. A process according to any one of the preceding claims wherein the intimate mixture of an oily or waxy substance and a nitrogen compound is added to the spray-dried powder by first spraying it onto a carrier material.

European Patent Office

EUROPEAN SEARCH REPORT

0087233
Application number

EP 83 30 0540

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. ³)
Y	DE-A-2 043 086 (HENKEL & CIE) * Claims 1, 2 *	1	C 11 D 3/32 C 11 D 3/26 C 11 D 3/00
Y	DE-A-2 043 088 (HENKEL & CIE) * Claims 1, 2 *	1	
D,Y	DE-A-2 043 087 (HENKEL & CIE) * Claims 1, 2 * & FR-A1-2 104 894	1	
A	EP-A-0 045 208 (UNILEVER PLC) * Claims 1, 3, 6, 7 *		
A	EP-A-0 021 830 (UNILEVER LTD) * Claims 8, 10 *		

TECHNICAL FIELDS SEARCHED (Int. Cl. ³)

C 11 D 3/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	08-04-1983	SCHULTZE D

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
O : non-written disclosure
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T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
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