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

(11) EP 0 709 449 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
01.05.1996 Bulletin 1996/18

(51) Int. Cl.[6]: **C11D 1/645**, C11D 1/52, C11D 11/00

(21) Application number: **94203141.0**

(22) Date of filing: **28.10.1994**

(84) Designated Contracting States:
AT BE CH DE DK ES FR GB GR IE IT LI LU NL PT SE

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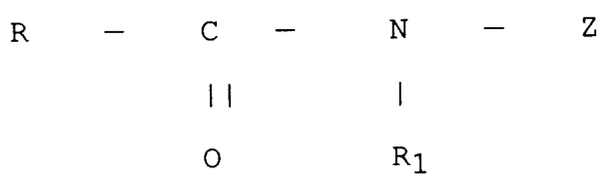
(54) Non-aqueous compositions comprising polyhydroxy fatty acid amides

(57) The invention concerns a concententrated liquid premix comprising polyhydroxy fatty acid amide of the formula:



where R is C5-C31 hydrocarbyl, preferably straight-chain C7-C19 alkyl or alkenyl,
R1 is C1-C8, preferably C1-C4 hydrocarbyl,
and Z is polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 2, preferably at least 3 hydroxyls directly connected to the chain,
wherein the liquid premix further comprises from 1% to 30% , preferably from 5% to 10%, by weight of choline ester.

EP 0 709 449 A1

Description

This invention concerns liquid premixes comprising certain nonionic surfactants, including fatty acid amides, which are suitable for further processing into detergent granules, especially for spraying on to prepared base granules to provide a finished granular detergent product.

The polyhydroxy fatty acid amides are one class of nonionic surfactants which are currently being investigated for use in detergent compositions. One problem with this class of surfactants is that concentrated aqueous solutions containing them tend to precipitate and/or gel on storage, even at elevated temperatures (35-60°). Moreover, low temperature storage of this family of amide surfactants is of great importance, since at elevated temperatures they are susceptible to degradation via hydrolysis of the amide bond to give the amine and the fatty acid. When polyhydroxy fatty acid amides are stored below 35°C this degradation is negligible, i.e. less than 5-10% per year, but at elevated temperatures it becomes highly significant, rising to about 10% per month at 50°C and about 20-25% per month at 60°C.

WO 9206160, published April 16th, 1992, describes aqueous compositions comprising polyhydroxy fatty acid amides and ethoxylated nonionic surfactants. Ratios between 1:5 and 5:1 are claimed.

One way to avoid problems of hydrolysis of the polyhydroxy fatty acid amide is to prepare non-aqueous premixes of polyhydroxy fatty acid amide. Non-aqueous premixes typically have high melting points (about 135°C for C16-18 glucose amide), and consequently for ease of processing it is necessary to depress the melting point of the amide by addition of suitable melting point depressants. Ethoxylated nonionic surfactants are suitable for this purpose if they are added at high levels, for example 2 parts ethoxylated nonionic to 1 part polyhydroxy fatty acid amide.

WO9000189, published January 11th, 1990, describes a process of spraying nonionic surfactant premix on to a base powder. It is claimed that solubility and dispersibility are improved if the nonionic premix comprises from 1% to 40% of hydrophobic substance containing polar groups (such as fatty acid, fatty acid ester, fatty acid amide, fatty amine, fatty alcohol or quaternary ammonium compound). There is no mention of problems associated with high melting points of any of the hydrophobic substances.

Furthermore, borate functional materials have been suggested as melting point depressants for both aqueoues and non-aqueous premixes.

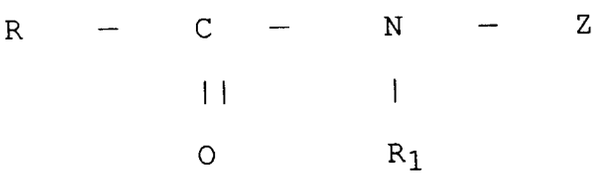
EP592754, published April 20th, 1994, describes a liquid premix comprising polyhydroxy fatty acid amide and a borate functional material. The benefit claimed is that the premix does not solidify or gel at temperatures above about 20-30°C. However borate functional materials may be unnecessary, or even undesirable in granular laundry detergents.

The present invention addresses the problem of providing an alternative melting point depressant for use in non-aqueous liquid premixes comprising polyhydroxy fatty acid amide. It has been found that choline ester acts to reduce the melting point of a typical polyhydroxy fatty acid amide / ethoxylated nonionic premix. The presence of 10% by weight of choline ester reduces the melting point by about 12°C. The possibility of storage and handling at lower temperatures has the further benefit of handling higher concentrations of polyhydroxy fatty acid amide than would be possible without the melting point depressant, and additionally reduces the rate of product decomposition.

Consequently, the present invention provides storage-stable, pumpable fluid compositions which contain relatively high concentrations of polyhydroxy fatty acid amide surfactants. Moreover, the invention provides such fluid compositions using ingredients which are either innocuous in the finished detergent composition, or which can provide desirable benefits to said finished compositions. Accordingly, removal of such ingredients is not required.

Summary of the Invention

The invention concerns a concententrated liquid premix comprising polyhydroxy fatty acid amide of the formula:



where R is C5-C31 hydrocarbyl, preferably straight-chain C7-C19 alkyl or alkenyl,
R1 is C1-C8, preferably C1-C4 hydrocarbyl,
and Z is polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 2, preferably at least 3 hydroxyls directly connected to the chain,
wherein the liquid premix further comprises from 1% to 30% , preferably from 5% to 10%, by weight of choline ester.

More preferably polyhydroxy fatty acid amide is C12-C18 linear alkyl N-methyl glucamine at a level of at least 25% by weight, even more preferably from 50% to 80% by weight, and most preferably from 60% to 75% by weight.

Generally, the molar ratio of the polyhydroxy fatty acid amide to the choline ester is from 20:1 to 2:1.

It is most preferred that the liquid premix is essentially anhydrous. Preferred embodiments may, however comprise from 5% to 50% by weight of ethoxylated nonionic surfactant, and may also optionally comprise from 1% to 40% by weight of a solvent chosen from propylene glycol, ethanol, glycerine or mixtures thereof.

The present invention also concerns a process of treating the liquid premix mentioned above to form granules by spraying the premix onto a preformed adsorbent detergent power.

Alternatively granules may be formed by agglomerating finely divided detergent powders in the presence of the premix, preferably in a high shear mixer.

Detailed Description of the Invention

By "concentrated liquid premix" herein is meant percentages of the polyhydroxy fatty acid amide typically in the range of at least 25% by weight, preferably 35% to 80% by weight, and most preferably from 45% to 75% by weight.

By "pumpable" herein is meant a viscosity below about 20 000 mPas, preferably below about 12 000 mPas.

"Viscosity" is measured by means of a Brookfield Viscometer Model DVII with a Thermosel System. The viscosity of the systems is measured at 80°C.

By "stable liquid premix" herein is meant a homogeneous, fluid, nonbirefringent liquid. This can be estimated visually using polarised light, and can be confirmed using a microscope under polarised light. There should be no crystallisation or precipitation when a sample is examined by the naked eye.

Polyhydroxy fatty acid amides :

Polyhydroxy fatty acid amides may be produced by reacting a fatty acid ester and an N-alkyl polyhydroxy amine. The preferred amine for use in the present invention is N-(R1)-CH2(CH2OH)4-CH2-OH and the preferred ester is a C12-C20 fatty acid methyl ester. Most preferred is the reaction product of N-methyl glucamine with C12-C20 fatty acid methyl ester.

Methods of manufacturing polyhydroxy fatty acid amides have been described in WO 92 6073, published on 16th April, 1992. This application describes the preparation of polyhydroxy fatty acid amides in the presence of solvents. In a highly preferred embodiment of the invention N-methyl glucamine is reacted with a C12-C20 methyl ester. It also says that the formulator of granular detergent compositions may find it convenient to run the amidation reaction in the presence of solvents which comprise alkoxylated, especially ethoxylated (EO 3-8) C12-C14 alcohols (page 15, lines 22-27). This directly yields nonionic surfactant systems which are preferred in the present invention, such as those comprising N-methyl glucamide and C12-C14 alcohols with an average of 3 ethoxylate groups per molecule.

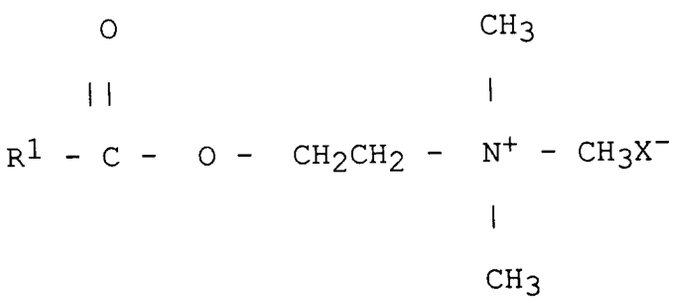
Nonionic surfactant systems, and granular detergents made from such systems have been described in WO 92 6160, published on 16th April, 1992. This application describes (example 15) a granular detergent composition prepared by fine dispersion mixing in an Eirich RV02 mixer which comprises N-methyl glucamide (10%), nonionic surfactant (10%).

Both of these patent applications describe nonionic surfactant systems together with suitable manufacturing processes for their synthesis, which have been found to be suitable for use in the present invention.

The polyhydroxy fatty acid amide may be present in compositions of the present invention at a level of from 0% to 40% by weight of the detergent component or composition, preferably from 1% to 30% by weight, even more preferably from 1% to 20% by weight.

Choline esters :

Preferred choline ester derivatives having the following formula :

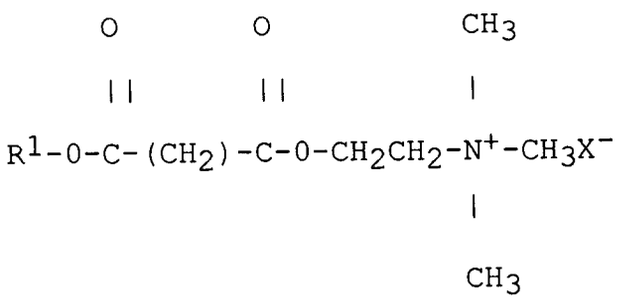


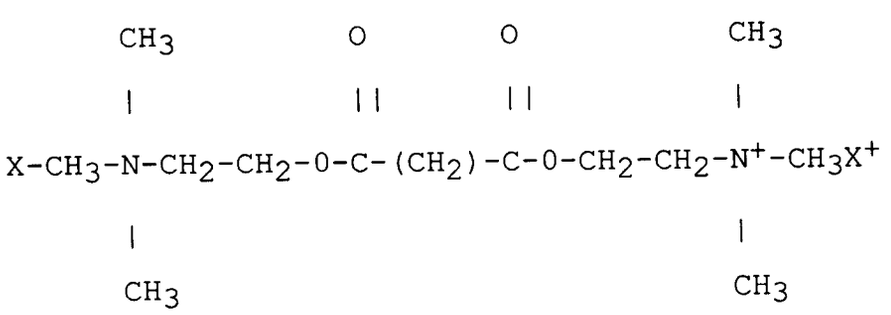
wherein R is a C_5 to C_{30} straight chain or branched chain alkyl or alkenyl, group and X is an anion, which makes the compound at least water-dispersible, preferably selected from the group consisting of halide, methyl sulfate, sulfate, and nitrate, preferably methyl sulfate, chloride, bromide or iodide.

as well as those wherein the ester linkage in the above formula is replaced with a reverse ester, amide or reverse amide linkage.

Particularly preferred examples of this type of cationic surfactant include stearoyl choline ester quaternary ammonium halides (R^1=C_{17} alkyl), palmitoyl choline ester quaternary ammonium halides (R^1=C_{15} alkyl), mystiroyl choline ester quaternary ammonium halides (R^1=C_{13} alkyl), lauroyl choline ester ammonium halides (R^1=C_{11} alkyl), as well as coconut and tallow choline ester quaternary ammonium halides (R^1=C_{15}-C_{17} alkyl and C_{19}-C_{13} alkyl, respectively).

Additional preferred cationic components of the choline ester variety are given by the structural formulas below, wherein p may be from 0 to 20.





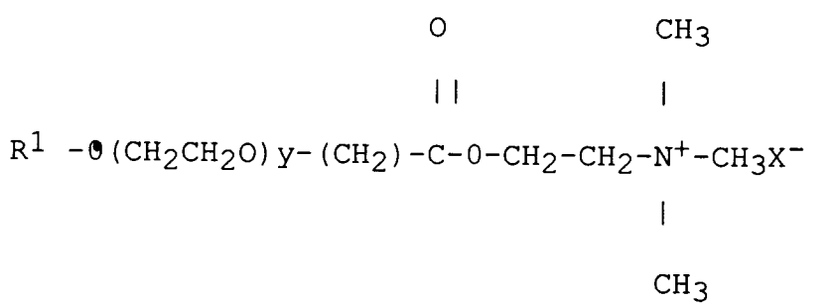
The preferred choline-derivative cationic substances, discussed above, may be prepared by the direct esterification of a fatty acid of the desired chain length with dimethylaminoethanol, in the presence of an acid catalyst. The reaction

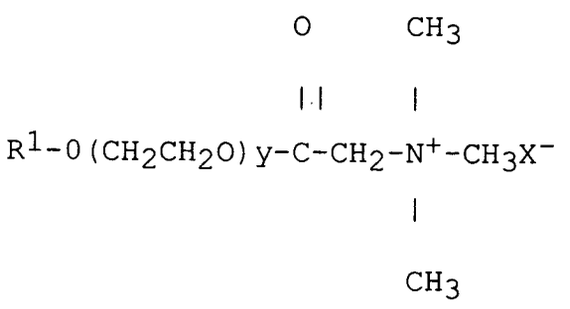
product is then quaternized with a methyl halide, forming the desired cationic material.

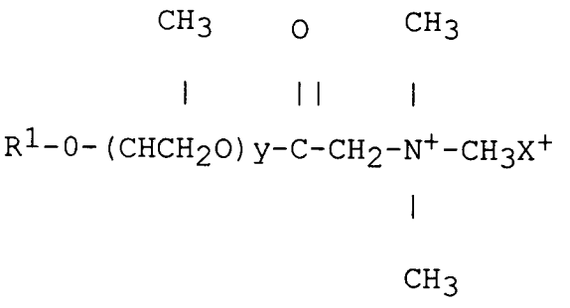
The choline-derived cationic materials may also be prepared by the direct esterification of a long chain fatty acid of the desired chain length together with 2-haloethanol, in the presence of an acid catalyst material. The reaction product is then used to quaternize.

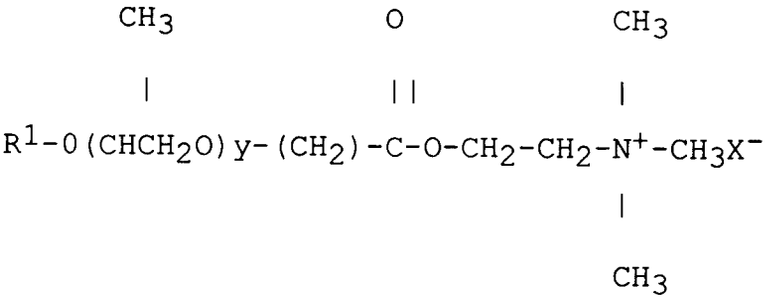
Trimethylamine, forming the desired cationic component.

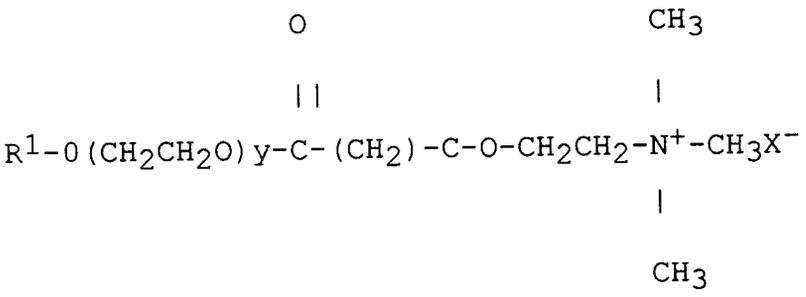
Other suitable choline esters for use herein have the formula:

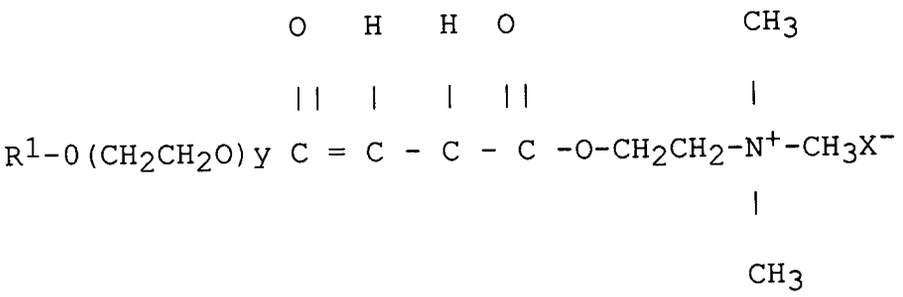


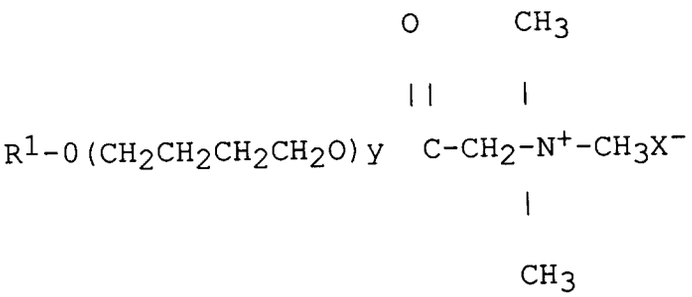


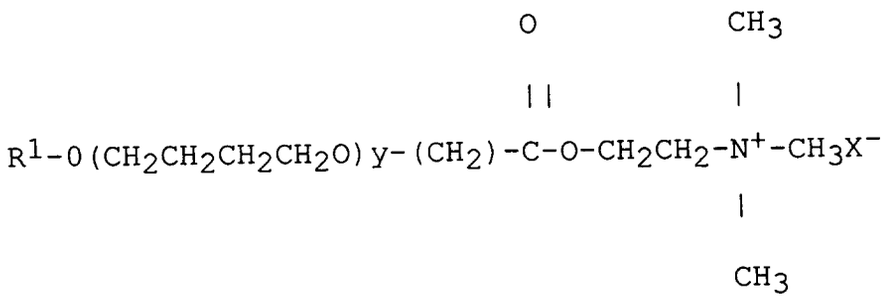












wherein t is 0 or 1, y is from 1 to 20, and R and X are as defined above.
The choline esters herein can be present at levels of from 0% to 50% by weight of the compositions, preferably from 1% to 30% by weight, even more preferably from 1% to 20% by weight.

Finished Product

The non-aqueous premixes of the present invention may be incorporated into granular laundry detergent compositions by any of the techniques known in the art. Granulation by agglomeration is one particularly preferred process. Spraying the premix onto a prepared base powder is an alternative preferred process.

Suitable agglomeration techniques are described in more detail in the Applicants co-pending European Application number 9401094.3, filed on 21st April 1994. In a highly preferrd agglomeration process the liquid premix of the present invention is intimately mixed with finely divided detergent powders, such as sodium aluminosilicate or other builders, in a high shear mixer, such as a Loedige® CB unit. Agglomerates may be finished in further mixing units, such as a Loedige® KM, or a fluidised bed.

Suitable techniques for spraying the premix onto prepared base powder as described in more detail in the Applicants co-pending Patent Application WO9405761, published on 17th March 1994.

EXAMPLES

In the following examples:
C16-18 N-methyl glucamide is CH_3 $(CH2)_n$ CO N CH_3 CH_2 $(CHOH)_4$ CH_2OH, where n=15-17
C12-15 alkyl ethoxylate (3EO) is CH_3 $(CH2)_m$ O $(CH_2CH_2O)_3$ H, where m=11-14
C12-14 Choline Ester is CH_3 $(CH2)_p$ COO $(CH_2)_2$ N $(CH_3)_3$ Cl, where p=11-13
The choline ester is handled in a propylene glycol solvent in a ratio of 7 parts choline ester to 3 parts propylene glycol (Examples 1 to 3) or 3 parts alkyl ethoxylate (Examples 4 to 6).

	Example 1	Example 2	Example 3
C16-18 N-methyl glucamide	27	36	45
C12-15 alkyl ethoxylate (3EO)	63	54	45
C12-14 Choline Ester	7	7	7
Propylene glycol	3	3	3
Melting Point* (°C)	67	64	63

*Melting Point is determined my means of Differential Scanning Calorimetry (DSC)

	Example 4	Example 5	Example 6
C16-18 N-methyl glucamide	27	36	45
C12-15 alkyl ethoxylate (3EO)	63	54	45
C12-14 Choline Ester	7	7	7
C12-14 Alkyl ethoxylate (3EO)	3	3	3
Melting Point* (°C)	67	65	63

*Melting Point is determined my means of Differential Scanning Calorimetry (DSC)

	Comparative Example A	Comparative Example B	comparative Example C
C16-18 N-methyl glucamide	30	40	50
C12-15 alkyl ethoxylate (3EO)	70	60	50
C12-14 Choline Ester	-	-	-
Propylene glycol	-	-	-
Melting Point* (°C)	74	75	75

*Melting Point is determined my means of Differential Scanning Calorimetry (DSC)

	Comparative Example D	Comparative Example E	Comparative Example F
C16-18 N-methyl glucamide	27	36	45
C12-15 alkyl ethoxylate (3EO)	63	54	45
C12-15 Choline Ester	-	-	-
Propylene glycol	10	10	10
Melting Point* (°C)	74	75	75

*Melting Point is determined my means of Differential Scanning Calorimetry (DSC)

Each of the liquid premix compositions of Examples 1 to 3 and A to F were prepared by mixing the raw materials in the appropriate ratios. The compositions were maintained at a temperature above the indicated melting point during transportation and storage. The lower storage temperatures of Examples 1 to 3, compared to Examples A to F, resulted in slower rates of degradation of the premix.

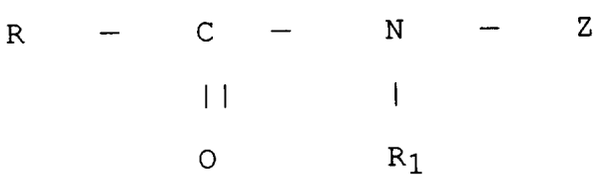
The liquid premix compositions were then sprayed on to a detergent base powder that had been prepared by agglomeration (of surfactants, builders etc.) and dry mixing (of granular percarbonate bleach, citric acid, granular suds suppressor, granular bleach activator, granular carbonate and bicarbonate, enzymes and encapsulated perfume). The ratio was 1 part liquid premix to 9 parts detergent base powder.

The temperature of the premix was reduced immediately before the spraying step to between 10° and 20°C below the indicated melting point so that crystallisation of the premix was starting to occur, but not to the extent that it would cause blockage of the nozzles. The spraying temperature was between 46°C and 50°C for Examples 1 to 3, and between 58°C and 62°C for Examples A to F (i.e a reduction of about 12°C in spraying on temperature).

The reduction of about 12°C in spraying on temperature made possible by the presence of the choline ester resulted in easier processing during the spraying on step. Examples 1 to 3 resulted in free-flowing detergent compositions. However Examples A to F resulted in compositions with a significant amount of lumps and oversize particles (Example C being worse than Example A and F being worse than example D). Finally Examples 1 to 3 showed better stability of the heat sensitive components, such as enzymes, than Examples A to F.

Claims

1. A liquid premix comprising polyhydroxy fatty acid amide of the formula:



where R is C5-C31 hydrocarbyl,
R1 is C1-C8 hydrocarbyl
and Z is polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 2 hydroxyls directly connected to the chain,
characterised in that the liquid premix further comprises from 1% to 30% by weight of choline ester.

2. A liquid premix according to claim 1 comprising polyhydroxy fatty acid amide wherein:
R is straight-chain C7-C19 alkyl or alkenyl,
R1 is C1-C4 hydrocarbyl
and Z is polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 3 hydroxyls directly connected to the chain,

3. A liquid premix according to claim 1 in which the choline ester is present at a level of from 5% to 10% by weight.

4. A liquid premix according to claim 1 in which the polyhydroxy fatty acid amide is C12-C18 linear alkyl N-methyl glucamine at a level of at least 25% by weight.

5. A liquid premix according to claim 4 in which the polyhydroxy fatty acid amide is C12-C18 linear alkyl N-methyl glucamine at a level of from 50% to 80% by weight, preferably from 60% to 75% by weight.

6. A liquid premix according to claim 5 in which the molar ratio of the polyhydroxy fatty acid amide to the choline ester is from 20:1 to 2:1.

7. A liquid premix according to claim 1 which is essentially anhydrous.

8. A liquid premix according to claim 7 further comprising from 5% to 50% by weight of ethoxylated nonionic surfactant.

9. A liquid premix according to claim 7, further comprising from 1% to 40% by weight of a solvent chosen from propylene glycol, ethanol, glycerine or mixtures thereof.

10. A process of treating the liquid premix of any of claims 1 to 9 to form granules by spraying the premix onto a preformed adsorbent detergent powder.

11. A process of treating the liquid premix of any of claims 1 to 9 to form granules by agglomerating finely divided detergent powders in the presence of the premix, preferably in a high shear mixer.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 94 20 3141

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.6)
D,A	WO-A-94 09099 (PROCTER & GAMBLE) 28 April 1994 * claims 1-3,7,8 *	1,2,4,5,9	C11D1/645 C11D1/52 C11D11/00
A	WO-A-93 19146 (PROCTER & GAMBLE) 30 September 1993 * claims 1-4 *	1,2,4,5,9	
D,A	WO-A-92 06073 (PROCTER & GAMBLE) 16 April 1992 * page 9, line 35 - page 10, line 2 * * page 23 - page 24; claims 1,8-10 *	1,2,4,5,9	
D,A	WO-A-94 05761 (PROCTER & GAMBLE) 17 March 1994 * page 8 - page 9 * * page 16 - page 19; claims 1-6 *	1,2,10,11	
D,A	WO-A-90 00189 (HENKEL KGAA) 11 January 1990 * abstract; claims *	1,10	

TECHNICAL FIELDS SEARCHED (Int.Cl.6)

C11D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	21 March 1995	Serbetsoglou, A

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
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