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(54) Titre: LIQUIDES STABLES A HAUTE TENEUR EN GLYCEROL COMPORTANT DES ACIDES N-ACYLAMINES OU LEUR SELS

(54) Title: STABLE, HIGH GLYCEROL LIQUIDS COMPRISING N-ACYL AMINO ACIDS OR SALTS THEREOF

(57) Abrégé/Abstract:

The present invention describes high content glycerol liquid compositions comprising N-acyl amino acids and/or salts and defined sulfosuccinic acid monoesters. Although large amounts of glycerin normally destabilizes such compositions, inclusion of dialkylene glycol has been found to enhance stability. A method of enhancing stability of high content glycerin compositions comprising N-acyl amino acids or salts thereof and sulfosuccinic acid monoesters is also described.





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(54) Title: STABLE, HIGH GLYCEROL LIQUIDS COMPRISING N-ACYL AMINO ACIDS AND/OR SALTS

(57) Abstract: The present invention describes high content glycerol liquid compositions comprising N-acyl amino acids and/or salts and defined sulfosuccinic acid monoesters. Although large amounts of glycerin normally destabilizes such compositions, inclusion of dialkylene glycol has been found to enhance stability. A method of enhancing stability of high content glycerin compositions comprising N-acyl amino acids or salts thereof and sulfosuccinic acid monoesters is also described.

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STABLE, HIGH GLYCEROL LIQUIDS COMPRISING N-ACYL AMINO ACIDS OR SALTS THEREOF

Field of the Invention

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The present invention relates to high content glycerol liquid compositions comprising N-acyl amino acids and/or salts thereof and also containing certain sulfosuccinic acid monoesters. More specifically, while high glycerin content has been found to destabilize such compositions, applicants have found that inclusion of dialkylene glycol stabilizes the compositions. Compositions of the invention are used primarily in skin cleansing, shower gel, and hair care compositions.

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Background of the Invention

Compositions containing N-acyl amino acids and/or salts and certain sulfosuccinic acid monoesters are beneficial to the skin. The use of both components together is broadly taught in U.S. Patent No. 4,749,515 to Miyamoto. Compositions of Miyamoto impart smoothness to the hair or skin after washing.

However, because of its skin moisturizing properties, it would be greatly beneficial to add glycerin to these or other compositions to help relieve dryness of skin. In the above compositions, however, it has been found that including large amounts of glycerin (e.g., about 10% and above, preferably 15% to 50% by wt., more preferably 20% to 45% by wt., more preferably 35% and above by wt.) led to

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instability and phase separation. While not wishing to be bound by theory, it is thought that this may be due to the high specific gravity of glycerin.

5 Unexpectedly, applicants have now found that if 0.5 to 25% by wt., preferably 1 to 15% by wt. of dialkylene glycol (e.g., dipropylene glycol) are used in the high content glycerin, N-acyl amino acid/sulfosuccinic acid monoester compositions, the compositions are stable (stability is maintained for at least 4 weeks at room temperature).

Brief Summary of the Invention

The present invention relates to stable high content glycerol liquid detergent compositions. More particularly, the compositions comprise:

- (1) 1-20%, preferably 5 to 20% by wt. of an N-acyl amino acid (e.g., N-acyl glycinate) or salts thereof;
- 20 (2) 0.5-15%, preferably 1-10%, by wt. of one or more sulfosuccinic acid monoesters represented by formula (I), (II), (III) or (IV);

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$$R_{2}CONHCH_{2}CHO(AO)_{n}$$
 C CH_{2} $M_{4}O$ C $CHSO_{3}M_{3}$ (II)

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$$R_1O(AO)_n$$
— C — $CHSO_3OM_1$
 M_2O — C — CH_2
(III)

or

$$R_{3}$$
 O R_{2} CONHCH₂CHO(AO)_n—C —CHSO₃M₃ (IV)

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wherein each of M_1 to M_4 is H, NH_4 , an alkali metal or a hydroxyalkyl substituted ammonium. R_1 and R_2 are each an alkyl or hydroxyalkyl group having about 8 to 20 carbon atoms, R_3 is H or CH_3 , AO is an oxyalkylene group having 2 or 3 carbon atoms, and n represents an integer from 0 to 20;

- (3) about 10% and above, preferably 15% to 50% by wt., more preferably 20% to 45% by wt., more preferably 35% and above by wt. of glycerin; and
- (4) 0 to 25% by wt., preferably 1 to 15% by wt. dialkylene glycol; and
- (5) water to balance (e.g., 5 to 90%, preferably 10 to 80%, more preferably 20 to 75% water).

In a second embodiment, the invention relates to a method of enhancing the stability of high content glycerin compositions comprising N-acyl amino acids or salts thereof and sulfosuccinic acid monoesters as defined above by adding 1 to 20% by wt. of dialkylene glycol.

The pH of the compositions is about 5.5 to 8, preferably 6 to 7.5 and the viscosity is about 300 to 10,000 centistokes.

Viscosity measurements were conducted using a Brookfield viscometer, Spindle No. 2 or 3, 12rpm, 30 seconds, at 25 degree C.

The stability of the composition is defined by the absence of separation after 4 weeks storage at room temperature.

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Detailed Description of the Invention

The present invention relates to stable, high content glycerol liquid detergent compositions used, for example, in skin cleansing, shower gel and/or shampoo compositions. More specifically, the compositions of the present invention comprise N-acyl amino acids and certain sulfosuccinic acid monoesters. Because of problems in maintaining stability, it has not been previously possible to make such compositions with high levels (i.e., equal to and above 10% up to 50% by wt. glycerol). Applicants have found, however, that addition of 0.5 to 25%, preferably 1 to 15% by wt. dialkylene glycol provides stability to such compositions. Compositions of the invention are described in greater detail below.

The compositions of the present invention contain about 1 to 20% by wt., preferably 5 to 20% by wt. of N-acyl amino acid and salts thereof.

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Acyl groups in the N-acyl amino acids and salts thereof which are suitable for the purposes of the present invention have 6 to 24 carbon atoms; for example, lauryl, myristyl, palmityl, or the like is included. The amino acids include glutamic acid, glycine, beta-alanine and the like. The salts include alkali metal salts, hydroxyalkyl substituted ammonium salts and ammonium salts. The hydroxyalkyl substituted ammonium salts may preferably have 1 to 3 carbon atoms in the hydroxyalkyl group. N-acyl-N-alkyl amino acids are also included in the term "N-acyl amino acids" used herein. The alkyl groups in the N-acyl-N-alkyl amino acids

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may preferably have 1 to 3 carbon atoms and include methyl, ethyl, propyl, isopropyl and the like. These N-acyl amino acids and salts thereof may be used independently or as a combination of two or more.

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Preferred N-acyl amino acids and salts thereof may include N-acyl amino acids such as N-lauroylglutamic acid, N-myristoylglutamic acid, N-palmitoyl-alpha-glutamic acid, N-myristoyl-beta-alanine, N-palmitoyl-beta-alanine and the like; N-acyl N-alkyl amino acids such as N-lauroyl-N-ethylglycine, N-lauroyl-N-isopropylglycine, N-lauroyl-N-isopropylglycine, N-palmitoylsarcosine, N-myristoylsarcosine, N-palmitoylsarcosine, N-lauroyl-N-methyl-beta-alanine and the like; as well as their alkali metal salts, hydroxyalkyl-substituted ammonium salts and the like.

The compositions of the present invention also comprise 0.5 to 15%, preferably 1-10% by wt. of a sulfosuccinic acid monoester represented by formulae (I), (II), (III) or (IV) or mixtures thereof as follows:

$$R_1O(AO)_n$$
 — C — CH_2 (I) M_2O — C — $CHSO_3M_1$

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$$R_{2}CONHCH_{2}CHO(AO)_{n}$$
 C CH_{2} $M_{4}O$ C $CHSO_{3}M_{3}$ (II)

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$$R_1O(AO)_n$$
— C — $CHSO_3OM_1$
 M_2O — C — CH_2
(III)

or

$$R_{2}CONHCH_{2}CHO(AO)_{n}$$
 — C — $CHSO_{3}M_{3}$ (IV)

 $M_{4}O$ — C — CH_{2}

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In these formulae, each of M_1 to M_4 may be selected from H, NH4, alkali metals and hydroxyalkyl-substituted ammonium M_1 and M_2 , and M_3 and M_4 may be the same or groups. different. Alkali metals may include lithium, sodium, 5 potassium and the like. The hydroxyalkyl substituted ammonium groups, which may preferably have 1 to 3 carbon atoms in the hydroxyalkyl group, may include monoethanol ammonium, diethanol ammonium, triethanol ammonium, methyl diethanol ammonium and the like. Preferably M₁ to M₄ are hydrogen, sodium or triethanol ammonium.

 R_1 and R_2 are each straight or branched alkyl or hydroxyalkyl groups having about 8 to 20 carbon atoms, for example, hexyl, decyl, hydroxydecyl, dodecyl, hydroxytetradecyl, 15 tetradecyl, nonadecyl, or the like. Any alkyl group having less than 8 or more than 20 carbon atoms may optionally be contained in the molecule, provided that the total number of carbon atoms in the sulfosuccinic acid monoester contained in the detergent composition of the present invention is in the range of from 8 to 20. This number of carbon atoms 20 provides good foaming properties, whereas when the number of carbon atoms is either less than 8 or more than 20 less foaming is observed.

In the formulae (II) and (IV), R3 is either H or CH3.

In the formulae, AO represents an oxyalkylene group having 2 or 3 carbon atoms, that is, oxyethylene or oxypropylene - 9 -

group. Both oxyethylene and oxypropylene groups may be present in the molecule.

The letter n represents an integer from 0 to 20, preferably 5 0 to 10. If n is more than 20, the foaming properties of the resulting liquid detergent compositions may be poor.

The compounds represented by the formulae (I), (II), (III) and (IV) may be prepared by any known methods.

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For example, the sulfosuccinic acid monoesters represented by the general formula (I) or (II) may be prepared by reacting an alkylene oxide adduct of a higher fatty acid with maleic anhydride to produce an ester of maleic anhydride and further reacting the ester of maleic anhydride with a sulfite.

Alternatively, the sulfosuccinic acid monoesters represented by the general formulae (II) or (IV) may be prepared by reacting a lower alcohol ester of a higher fatty acid with an alkanolamine, adding an alkylene oxide to the reaction product, further reacting the resulting addition product with maleic anhydride to produce an ester of maleic anhydride, and reacting the ester of maleic anhydride with a 25 sulfite.

The compositions of the present invention also comprise about 10% and above, preferably 15% to 50% by wt, more preferably 20% to 45% by wt. of glycerin.

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Finally, the compositions of the invention also comprise 1-20%, preferably 2-15% by wt. of a polyalkylene glycol, preferably dialkylene glycol.

5 Especially preferred are dialkylene glycol molecules such as, for example, diethylene or dipropylene glycol.

As noted above, it is the specific inclusion of such polyalkylene glycol molecules which is believed to stabilize the high content glycerin compositions of the present invention.

The compositions of the present invention may also contain many optional components as set forth below.

Specifically, in addition to the glycinate of the invention, compositions of the invention may contain 0.1 to 10% by wt. of an additional surfactant or surfactants selected from anionic, nonionic, amphoteric and cationic surfactants and mixtures thereof.

It is especially desired to contain at least 0.10 to 10% by wt. of an additional anionic surfactant.

Suitable anionic detergents include, but are not limited to, alkyl and alkylene carboxylates having 10 to 20 carbon atoms, alkyl ether carboxylates, fatty alcohol sulfates, fatty alcohol ether sulfates, alkylol amido-sulfates and sulfonates, fatty acid alkylol amido-polyglycol ether sulfates, alkane sulfonates and hydroxyalkane sulfonates, olefin sulfonates, acyl esters of isethionates, α -sulfo

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fatty acid esters, alkyl-benzene sulfonates, alkylphenol glycol ether sulfonates, sulfo-succinates, sulfosuccinic acid half and di-esters, fatty alcohol ether phosphates, protein fatty acid condensation products, alkyl monoglyceride sulfates and sulfonates, alkyl glyceride ether sulfonates, fatty acid methyl taurides, fatty acid sarcosinates, sulfo-ricinoleates. These compounds and mixtures thereof are used in the form of their water-soluble or water-dispersible salts, for example the sodium, potassium, magnesium, ammonium, mono-, di- and triethanolammonium salts and analogous alkylol ammonium salts.

particularly preferred anionic surfactant is polyoxyalkylene alkylether sulfate which maybe used in an amount of, for example, 0.01 to 5% by wt.

nonionic surface-active agents include, for Suitable example, fatty alcohol ethoxylates (alkyl-polyethylene glycols), alkylphenol-polyethylene glycols, alkylationpolyethylene glycols, fatty amine ethoxylates (alkylaminopolyethylene glycols), fatty acid ethoxylates (acyl polyethylene glycols), polypropylene glycol ethoxylates (Pluronic (Registered Trademark)), fatty acid alkyl amides (fatty acid amino polyethylene glycols), saccharose esters, sorbitol esters, and polyglycol ether. 25

Suitable amphoteric surface-active agents to be added to the shampoos include N-alkyl- β -amino dipropionates having 12 to 18 alkyl carbon atoms as alkali metal salts and mono-, diand trialkylol-ammonium salts, N-acylamido-alkyl-N, Ndimethylacetobetaine, preferably N-acyl-amidopropyl-N,N-

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dimethylacetobetaine of 8 to 18 acyl carbon atoms, alkyldimethylsulfopropyl-betaine having 12 to 18 alkyl carbon atoms, amphoteric surfactants of the imidazoline type (Trademarks: Miranol Steinapon), preferably the sodium salt of $1-(\beta-\text{carboxy-methyloxethyl})-1-(\text{carboxymethyl})-2-lauryl-imidazolinium; amine oxides, for example alkyldimethyl-amine oxide of 12 to 18 alkyl carbon atoms, fatty acid amidoalkyl-dimethylamine oxide.$

10 A particularly preferred amphoteric is cocoamido propyl betaine.

Among suitable cationics are included, for example quaternary ammonium salts, such as dialkyl-dimethyl15 ammonium, chloride or bromide having 10 to 24, preferably 12 to 18 carbon atoms in the alkyl portion, alkyl-dimethylethylammonium chloride or bromide having 10 to 24 alkyl carbon atoms, alkyl-trimethyl-ammonium chloride or bromide having 10 to 24 alkyl carbon atoms, preferably cetyl20 trimethyl ammonium chloride or bromide, alkyl-trimethyl ammonium chloride or bromide having 10 to 22 alkyl carbon atoms, or alkyl-dimethyl-benzyl ammonium chloride or bromide having 10 to 24, preferably 12 to 18, carbon atoms in the alkyl portion.

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In addition to the ingredients noted above, a variety of optional ingredients may be included in the compositions of the present invention.

Preferred optional ingredients are free fatty acids, i.e., $C_{10}-C_{24}$, preferably C_{12} to C_{18} straight chained, preferably

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saturated fatty acids. Examples include lauric and/or myristic acid. These should be used in an amount from about 0.1 to 10%, preferably 2 to 8% by wt. While not wishing to be bound by theory, the fatty acids are believed to enhance stability.

The liquid personal cleansing compositions of the present invention may optionally also include water-dispersible, gel-forming polymers. The polymer is preferably a anionic, nonionic, cationic or hydrophobically modified polymer, selected from cationic polysaccharides of the cationic quar gum class with molecular weights of 1,000 to 3,000,000, anionic, cationic and nonionic homopolymers derived from acrylic and/or methacrylic acid, anionic, cationic and nonionic cellulose resins; cationic copolymers of dimethyldialkyl ammonium chloride and acrylic acid; cationic homopolymers of dimethyldialkyl ammonium chloride; cationic polyalkylene and ethoxypolyalkylene imines polyethylene glycol with molecular weights of 10,000 to 4,000,000; and mixtures thereof. Preferably, the polymer is selected from Sodium Polyacrylate, Hydroxy Ethyl Cellulose, Cetyl Hydroxy Ethyl Cellulose, and Polyquaternium 10.

The polymer is preferably included in the compositions of the present invention at a level of from apout 0.1 parts to 1 part, more preferably 0.1 parts to 0.5 parts. The polymers improve the sensory feel of the lipid on skin in addition to providing product stabilization. The improved sensory feel results from reduced tackiness and greasiness and improved smoothness. In an especially preferred embodiment a mixture of polymers is used, for example, those

polymers preferred for product stabilization, some are preferred for improved sensory feel and/or those preferred polymers for improved sensory feel. Preferred polymers for improved sensory feel are selected from polyethylene glycol, hydroxypropyl guar, guar hydroxypropyl trimonium chloride, polyquaternary 3, 5, 6, 7, 10, 11 and 24 and mixtures thereof.

The balance of the composition (generally between about 10 to 70%, preferably 20 to 60% by wt.) is water.

A variety of additional ingredients may be incorporated into the compositions of the present invention. These materials include, but not limited to, liquid appearance aids, salts and their hydrates and other "filler materials" are listed in U.s. Patent No. 5,340,492 to Kacher et al., and U.S. Patent No, 4,919,934 to Deckner et al.

Other non-limiting examples of these additional ingredients include vitamins and derivatives thereof (e.g., ascorbic acid, vitamin E, tocopherol acetate, and the like); sunscreens; thickening agents (e.g., polyol alkoxy ester, available as Crothix^m from Croda at levels up to 2% and 25 xanthan gum at levels up to 2%); preservatives for maintaining the anti-microbial integrity of the compositions; anti-acne medicaments (resorcinol, salicylic acid, and the like); antioxidants; skin soothing and healing agents such as aloe vera extract, allantoin and the like; 30 chelators (e.g., EDTA and hydroxy ethan diphosphoric acid) and sequestrants; and agents suitable for aesthetic

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purposes such as fragrances, essential oils, skin sensates, pigments, pearlescent agents (e.g., mica and titanium dioxide), additives to impart a draggy rinse feel (e.g., fumed silica), additives to enhance deposition (e.g., maleated soybean oil at levels up to 3%), lakes, colorings, and the like (e.g., clove oil, menthol, camphor, eucalyptus oil and eugenol).

In a second embodiment, the invention relates to a method of enhancing the stability of high glycerin content compositions containing N-acyl amino acids or salts thereof and defined sulfosuccinic acid monoesters by adding 1 to 20% of polyalkylene glycol, preferably dialkylene glycol, as noted above.

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Compositions of the present invention have a pH of 5.5 to 8, preferably 6 to 7.5.

Compositions of the present invention have a viscosity of 300-10,000 centistokes measured using a Brookfield Viscometer, Spindle 2 and 3 at 12 rpm for 30 seconds at 25°C.

The compositions of the present invention exhibit no precipitation or phase separation after 4 weeks storage at room temperature (i.e., about 25°C).

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EXAMPLES

Example 1 and 2

5 The following are two examples of the compositions of the invention.

	Example 1	Example 2
Sodium N-cocoyl glycinate(SCG)	10%	10%
Disodium laurylether sulfosuccinate(DSLES)	2%	2%
Disodium lauryl sulfosuccinate(DSLS)	2%	2 %
Lauric acid	3%	3%
Myristic acid	2%	2%
Potassium hydroxide	0.67%	0.67%
Sodium laurylether sulfate(SLES)	0.88%	0.88%
Cocamidopropyl betaine(CAPB)	0.12%	0.12%
Ethylene glycol distearate(EGDS)	3%	3%
Dipropylene glycol(DPG)	3%	3%
Glycerin	37%	20%
Amphoteric polymer	0.3%	0.3%
Alkyl acrylate polymer	0.2%	0.5%
Tetrasodiumedetate tetrahydrate(EDTA)	0.05%	0.05%
Dibutylhydroxtoluene(BHT)	0.05%	0.05%
Preservative	0.3%	0.3%
Perfume	0.7%	0.7%
Water	To 100%	To 100%

The compositions noted above were prepared as follows:

Processing

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1) A mixture of mostly glycerin and SCG was heated to $75^{\circ}-80^{\circ}\text{C}$ until becoming quite fluid (mixture-1);

- 2) A mixture of fatty acids, preservative, DPG, BHT and SLES was heated to $75^{\circ}-80^{\circ}$ C until becoming fluid (mixture-2);
- 3) Mixture-2 was added to mixture-1 with
 agitation(mixture-3);
- 4) A mixture of potassium hydroxide and water was heated to $75^{\circ}-80^{\circ}$ C (mixture-4);
- 5) Mixture-4 was added to mixture-3 with agitation (mixture-5);
- 10 6) CAPB, DSLES, DSLS and EGDS were separately added to mixture-5 with agitation (mixture-6);
 - 7) Mixture-6 was cooled to 45°C;
 - 8) Water and EDTA were mixed and then added to mixture-6 (mixture-7);
- 9) Water and amphoteric polymer were mixed and then added to mixture-7 (mixture-8);
 - 10) Glycerin and alkyl acryl polymer were mixed and then added to mixture-8 (mixture-9);
 - 11) Perfume was added to mixture-9 and cooled to 35°C.

Example 3

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In a composition containing DPG and glycerin the following data was observed in a composition as set forth in Example 1 (levels of glycerin were offset by water).

	<u>DPG</u>	Glycerin	RT stability
	0 %	40%	Separation; no good
	3%	37%	Acceptable level; good
30	5%	35%	Acceptable level; good

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10%	30%	Acceptable level; slightl	slightly	
		good		
0 응	30%	Separation; no good		

5 The data above clearly shows that defined levels of dialkylene glycol enhance stability of high glycerin content compositions containing N-acyl amino acid and sulfosuccinic acid monoesters while, in their absence, phase separation occurs.

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CLAIMS

- 1. A stable, high glycerol liquid detergent composition comprising:
- (a) 1-20% by wt. N-acyl amino acid or salt of such acid;
 - (b) 0.5-15% by wt. of one or more sulfosuccinic acid
 monoesters represented by Formula (I), (II),
 (III) or (IV):

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$$R_1O(AO)_n$$
 — C — CH_2 (I) M_2O — C — $CHSO_3M_1$ C

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$$R_{2}CONHCH_{2}CHO(AO)_{n} - C - CH_{2}$$

$$M_{4}O - C - CHSO_{3}M_{3}$$
(II)

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$$R_{1}O(AO)_{n} - C - CHSO_{3}OM_{1}$$

$$M_{2}O - C - CH_{2}$$
(III)

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$$R_3$$
 O R_2 CONHCH₂CHO(AO)_n — C — CHSO₃M₃ (IV)

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wherein each of M₁ to M₄ is H, NH₄, an alkali metal or a hydroxyalkyl substituted ammonium, R₁ and R₂ are each an alkyl or hydroxyalkyl group having 8 to 20 carbon atoms, R₃ is H or CH₃, AO is an oxyalkylene group having 2 or 3 carbon atoms, and n represents an integer from 0 to 20;

- (c) 10% and above by wt. of glycerin;
- 25 (d) 1-20%, by wt. of dialkylene glycol; and
 - (e) balance water.

- 2. A composition according to claim 1, wherein the composition additionally comprises 0.01-10% by weight of a surfactant selected from anionic surfactants, nonionic surfactants, amphoteric surfactants, cationic surfactants and mixtures thereof.
- 3. A composition according to claim 2, wherein the amphoteric surfactant is a betaine.
- 4. A composition according to any one of claims 1 to 3, wherein the composition additionally comprises 0.01 to 10% by weight free fatty acid.
- 5. A composition according to any one of claims 1 to 4, wherein the composition has a pH of 5.5 to 8.
- 6. A composition according to claim 1, wherein the composition has a viscosity of 300 to 10,000 centistokes at 25°C.
- 7. A composition according to claim 1, having the characteristic that no precipitation and no phase separation is exhibited after 4 weeks at room temperature.