

(19) World Intellectual Property
Organization
International Bureau



(43) International Publication Date
4 March 2004 (04.03.2004)

PCT

(10) International Publication Number
WO 2004/018601 A1

- (51) International Patent Classification⁷: **C11D 3/22**, 17/00, 1/94, 3/02, A61K 7/00, 7/50 // C11D 1:29, 1/14, 1/52 // 1/90
- (21) International Application Number: PCT/EP2003/008590
- (22) International Filing Date: 1 August 2003 (01.08.2003)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data: 10/218,236 14 August 2002 (14.08.2002) US
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- (81) Designated States (national): AE, AG, AL, AM, AT (utility model), AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ (utility model), CZ, DE (utility model), DE, DK (utility model), DK, DM, DZ, EC, EE (utility model), EE, ES, FI (utility model), FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK (utility model), SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
- Published:
— with international search report
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



WO 2004/018601 A1

(54) Title: BIPHASIC COMPOSITION INDUCED BY POLYDEXTROSE

(57) Abstract: The invention relates to use of specifically defined polydextrose to induce biphasic liquid formation. Optional use of salt allows less polydextrose to be used to induce biphasic formation.

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BIPHASIC COMPOSITION INDUCED BY POLYDEXTROSE

The present invention relates to aqueous liquid cleansing compositions which are biphasic in nature. More
5 specifically, such compositions are characterized by having (assuming they have been standing a sufficiently long period of time after shaking) both an upper aqueous layer and a separate lower aqueous layer. In the subject invention, formation of the biphasic liquids is induced by use of
10 sufficient amount of polydextrose wherein the polydextrose is within an approximate molecular weight window. The amount of polydextrose required to induce biphasic formation can be reduced if some salt is used.

15 Biphasic liquids defined by the general fact that the liquid is divided into two phases are not new. Most of these liquids comprise both one layer which is an aqueous layer, and a second layer comprising a water immiscible oily material.

20

U.S. Patent No. 3,718,609 to Weimer, for example, discloses a liquid detergent composition having an aqueous layer and a layer of liquid water immiscible oily material. When shaken, the liquid forms a temporary oil-in-water emulsion.

25

Similarly, U.S. Patent No. 3,810,478 to Olson Jr. et al. discloses a two phase shampoo composition made by preparing substantially polar and lipophilic portions of a shampoo composition.

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Biphasic compositions comprising an upper and lower aqueous phase are also disclosed in the art. U.S. Serial No. 09/643,142 to Williams et al., entitled "A Separating Multi-Phase Personal Wash Composition in a Transparent or Translucent Package" discloses biphasic compositions comprising:

- (a) 5 % to 35 % surfactant;
- (b) 1 % to 12 % thickener;
- (c) 4 % to 20 % polyalkylene glycol; and
- 10 (d) sufficient non-chelating mineral salt to induce phase separation.

While the total amount of salt/electrolyte is not specifically discussed in the above specification, it is apparent from the examples that the amount sufficient to induce formation of biphasic layer is at least in the order of 4 %, 5 %, 6 % and greater. By contrast, in the subject invention, salt is not required at all for biphasic formation and, if used, is generally in an amount less than 3 %, preferably about 2 % by wt. or less and more preferably about 1 % by wt. or less. As discussed in the specification below, using small amounts of salt (i.e., about 0.5 % to 3 %, preferably 0.5 % to 1 %) does allow a lesser amount of polydextrose to be used to induce biphasic formation.

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In addition, unlike the compositions in the Williams et al. specification, the compositions of the subject invention are induced by polydextrose, and are stable and they do not require either thickener or polyalkylene glycol as is required by the compositions of Williams.

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EP-A-0,116,422 to Reckett and Coleman also discloses multi-layered compositions in which two liquids are dispersible, and which separate on standing. Again, at least 6 % salt/electrolyte (e.g., sodium hexamataphosphate) are
5 required in these compositions (see page 4, lines 17-19). The biphasic liquids of the invention are induced by polydextrose, not salt, and no salt is required, although small amounts (e.g., up to about 3 %, preferably about 2 % or less, more preferably about 1.5 % or less, more
10 preferably about 1 % or less) may be used.

In addition, the compositions of the subject invention are preferably used in translucent or transparent compositions (i.e., for the sensorial benefit) and such is not taught or
15 suggested in EP 0,116,422.

Unexpectedly, the applicants have now found that biphasic liquids (e.g., liquids which separate into top and bottom aqueous liquids) may be induced merely by addition of
20 sufficient quantity of specifically defined polydextrose.

More specifically, the present invention comprises liquid personal cleansing compositions comprising:

- (1) 5 % to 75 %, preferably 6 % to 40 % by wt. of a
25 surfactant selected from the group consisting of anionic surfactants, nonionic surfactants, amphoteric/zwitterionic surfactants, cationic surfactants and mixtures thereof;
- (2) at least about 15 %, more preferably at least 20 %
30 by wt. of polydextrose or mixture of polydextrose molecules, wherein the degree of polymerization

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(e.g., number of linking glucose units) is 4 to 22 (this corresponds to MW of about 600 to about 3600); and

(3) balance water and minors.

5

In a second embodiment of the invention, the invention comprises compositions wherein at least 1 % salt is used and levels of polydextrose may be 10 %. In a further embodiment, the invention comprises composition wherein at least 2 % salt is used and polydextrose level may be as low as 5 %.

The present invention relates to biphasic liquid cleansing compositions wherein the formation of the biphasic liquid is induced by the addition of sufficient amount of polydextrose, wherein the degree of polymerization (defining number of linking glucose groups) is 4 to 22. This corresponds approximately to MW of about 600 to 3600.

20 More specifically, the invention comprises:

(1) 5 % to 75 % by wt. of a surfactant selected from the group consisting of anionic surfactant, nonionic surfactants, amphoteric/zwitterionic, cationic surfactant and mixtures thereof;

25 (2) at least 15 % polydextrose, wherein the degree of polymerization (i.e., number of linking glucose units) is 4 to 22 or have an MW of 600 to 3600; preferably MW is 700 to 1800, more preferably 900 to 1500 and more preferably 900 to 1200; and

30 (3) balance water and minors.

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The general concept behind the invention is that, when sufficient amount of specified polydextrose is added, phase separation occurs. For example, this is shown in the Examples wherein, when 20 % polydextrose MD180 (MW 1000) is added, separation occurs. Different surfactant systems can be used, and the specific type of surfactant is not a limiting factor.

The inventive compositions may be used in combination with a transparent package in order to view the liquid. Thus, in one embodiment, the invention also comprises a system comprising said transparent or translucent package in combination with the liquid.

Typically, once the biphasic composition is formed (e.g., the composition "settles" after having been shaken), the viscosity of the lower layer is lower than that of the upper layer.

Also, the density of lower layer is typically greater than that of upper layer.

Typically, in such biphasic liquids, there is no recrystallization visible after composition has been standing for 6 months at room temperature.

The final product will have shower-gel like viscosity of 100 to 5000 mPas, preferably 200 to 4000 at shear rate $10s^{-1}$ at 25°C measured using Haake RV20 Rotovisco Rheometer.

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In a second embodiment of the invention, a small amount of salt is used and the amount of polydextrose needed to induce biphasic liquid is reduced. More specifically, in this embodiment, the composition comprises at least 1 % salt and
5 at least 10 % polydextrose.

In a third embodiment, the composition comprises at least 2 % salt and at least 5 % polydextrose.

10 The various components of the composition are discussed in greater detail below.

The surfactant generally will comprise 5 % to 75 % by wt. of the total composition.

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The surfactant is a surfactant which may be selected from the group consisting of anionic surfactants, nonionic surfactants, amphoteric/zwitterionic surfactants, cationic surfactants and mixtures thereof. Preferably, there will be
20 at least one anionic surfactant.

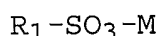
Non-limiting examples of anionic surfactants are disclosed in McCutcheon's Detergents and Emulsifiers, North American Edition (1986), published by Allured Publishing Corporation;
25 McCutcheon's Functional materials, North Americas Edition (1992), both of which are incorporated by reference into the subject application.

Examples of anionic surfactants include sarcosinates,
30 sulfates, isethionates, taurates, phosphates, lactylates, glutamates and mixtures thereof. Among isethionates are

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preferred alkoxy isethionates such as sodium cocoyl isethionate, sodium lauroyl isethionate and mixtures.

The alkyl and alkyl ether sulfates typically have the
5 respective formulae $ROSO_3M$ and $RO(C_2H_4O)_xSO_3M$, wherein R is alkyl or alkenyl of from about 10 to about 30 carbon atoms, x is from about 1 to about 10, and M is a water-soluble cation such as ammonium, sodium, potassium, magnesium and triethanolamine. Another suitable class of anionic
10 surfactants are the water-soluble salts of the organic, sulfuric acid reaction products of the general formula:



15 wherein R_1 is chosen from the group consisting of a straight or branched chain, saturated aliphatic hydrocarbon of radical having from about 8 to about 24, preferably about 10 to about 16, carbon atoms; and M is a cation. Still other anionic synthetic surfactants include the class designated
20 as succinamates, olefin sulfonates having about 12 to about 24 carbon atoms, and β -alkyloxy alkane sulfonates. Examples of these materials are sodium lauryl sulfate and ammonium lauryl sulfate.

25 Other anionic materials useful herein are soaps (i.e., alkali metal salts, e.g., sodium or potassium salts or ammonium or triethanolamine salts) of fatty acids, typically having from about 8 to about 24 carbon atoms, preferably from about 10 to about 20 carbon atoms. The fatty acids
30 used in making the soaps can be obtained from natural

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sources such as, for instance, plant or animal-derived glycerides (e.g., palm oil, coconut oil, soybean oil, castor oil, tallow, lard, etc.). The fatty acids can also be synthetically prepared. Soaps are described in more detail
5 in U.S. Patent No. 4,557,853.

Other useful anionic materials include phosphates such as monoalkyl, dialkyl, and trialkylphosphate salts.

10 Other anionic materials include alkanoyl sarcosinates corresponding to the formula $RCON(CH_3)CH_2CH_2CO_2M$ wherein R is alkyl or alkenyl of about 10 to about 20 carbon atoms, and M is a water-soluble cation such as ammonium, sodium, potassium and alkanolamine (e.g., triethanolamine), a
15 preferred examples of which are sodium lauroyl sarcosinate, sodium cocoyl sarcosinate, ammonium lauroyl sarcosinate, and sodium myristoyl sarcosinate. TEA salts of sarcosinates are also useful.

20 Also useful are taurates which are based on taurine, which is also known as 2-aminoethanesulfonic acid. Especially useful are taurates having carbon chains between C_8 and C_{16} . Examples of taurates include N-alkyltaurines such as the one prepared by reacting dodecylamine with sodium isethionate
25 according to the teaching of U.S. Patent No. 2,658,072 which is incorporated herein by reference in its entirety. Further non-limiting examples include ammonium, sodium, potassium and alkanolamine (e.g., triethanolamine) salts of lauroyl methyl taurate, myristoyl methyl taurate, and cocoyl
30 methyl taurate.

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Also useful are lactylates, especially those having carbon chains between C₈ and C₁₆. Non-limiting examples of lactylates include ammonium, sodium, potassium and alkanolamine (e.g., triethanolamine) salts of lauroyl lactylate, cocoyl lactylate, lauroyl lactylate, and caproyl lactylate.

Also useful herein as anionic surfactants are alkylamino carboxylates such as glutamates, especially those having carbon chains between C₈ and C₁₆. Non-limiting examples of glutamates include ammonium, sodium, potassium and alkanolamine (e.g., triethanolamine) salts of lauroyl glutamate, myristoyl glutamate, and cocoyl glutamate.

Non-limiting examples of preferred anionic lathering surfactants useful herein include those selected from the group consisting of sodium lauryl sulfate, ammonium lauryl sulfate, ammonium laureth sulfate, sodium laureth sulfate, sodium trideceth sulfate, ammonium cetyl sulfate, sodium cetyl sulfate, ammonium cocoyl isethionate, sodium lauroyl isethionate, sodium lauroyl lactylate, triethanolamine lauroyl lactylate, sodium caproyl lactylate, sodium lauroyl sarcosinate, sodium myristoyl sarcosinate, sodium cocoyl sarcosinate, sodium lauroyl methyl taurate, sodium cocoyl methyl taurate, sodium lauroyl glutamate, sodium myristoyl glutamate, and sodium cocoyl glutamate and mixtures therefor.

Especially preferred for use herein is ammonium lauryl sulfate, ammonium lauryl ether sulfate, sodium lauryl ether

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sulfate, sodium lauroyl sarcosinate, sodium cocoyl sarcosinate, sodium myristoyl sarcosinate, sodium lauroyl lactate, and triethanolamine lauroyl lactylates.

5 Non-limiting examples of nonionic lathering surfactants for use in the compositions of the present invention are disclosed in McCutcheon's, Detergents and Emulsifiers, North American Edition (1986), published by allured Published Corporation; and McCutcheon's, Functional materials, North
10 American Edition (1992); both of which are incorporated by reference herein in their entirety.

Nonionic lathering surfactants useful herein include those selected form the group consisting of alkyl glucosides,
15 alkyl polyglucosides, polyhydroxy fatty acid amides, alkoxyated fatty acid esters, alcohol ethoxylates, lathering sucrose esters, amine oxides, and mixtures thereof.

20 Alkyl glucosides and alkylpolyglucosides are useful herein, and can be broadly defined as condensation articles of long chain alcohols, e.g., C8-30 alcohols, with sugars or starches or sugar or starch polymers i.e., glycosides or polyglycosides. These compounds can be represented by the
25 formula $(S)_n-O-R$ wherein S is a sugar moiety such as glucose, fructose, mannose, and galactose; n is an integer of from about 1 to about 1000, and R is a C8-30 alkyl group. Examples of long chain alcohols from which the alkyl group can be derived include decyl alcohol, cetyl alcohol, stearyl
30 alcohol, lauryl alcohol, myristyl alcohol, oleyl alcohol and the like.

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Preferred examples of these surfactants include those wherein S is a glucose moiety, R is a C8-20 alkyl group, and n is an integer of from about 1 to about 9. Commercially available examples of these surfactants include decyl
 5 polyglucoside (available as APG 325 CS from Henkel) and lauryl polyglucoside (available as APG 600 CS and 625 CS from Henkel). Also useful are sucrose ester surfactants such as sucrose cocoate and sucrose laurate.

10 Other useful nonionic surfactants include polyhydroxy fatty acid amide surfactants, more specific examples of which include glucosamides, corresponding to the structural formula:



wherein R¹ is H, C₁-C₄ alkyl, 2-hydroxyethyl, 2-hydroxypropyl, preferably C₁-C₄ alkyl, more preferably methyl or
 20 ethyl, most preferably methyl; R² is C₅-C₃₁ alkyl or alkenyl, preferably C₇-C₁₉ alkyl or alkenyl, more preferably C₉-C₁₇ alkyl or alkenyl, most preferably C₁₁-C₁₅ alkyl or alkenyl; and Z is a polyhydroxy hydrocarbyl moiety having a linear hydrocarbyl chain with at least 3 hydroxyl directly
 25 connected to the chain, or an alkoxyated derivative (preferably ethoxyated or propoxyated) thereof. Z preferably is a sugar moiety selected from the group consisting of glucose, fructose, maltose, lactose, galactose, mannose, xylose, and mixtures thereof.

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As especially preferred surfactant corresponding to the above structure is coconut alkyl N-methyl glucoside amide (i.e., wherein the R²CO-moiety is derived from coconut oil fatty acids). Processes for making compositions containing polyhydroxy fatty acid amides are disclosed, for example, in 5 GB Patent Specification 809,060, published February 18, 1959, by Thomas Hedley & Co., Ltd.; U.S. Patent No. 2,965,576, to E.R. Wilson, issued December 20, 1960; U.S. Patent No. 2,703,798 to A. M. Schwartz, issued March 8, 10 1955; and U.S. Patent No. 1,985,424, to Piggott, issued December 25, 1934; which are incorporated herein by reference in their entirety.

Other examples of nonionic surfactants include amine oxides. 15 Amine oxides correspond to the general formula R₁R₂R₃NO, wherein R₁ contains an alkyl, alkenyl or monohydroxyl alkyl radical of from about 8 to about 18 carbon atoms, from 0 to about 10 ethylene oxide moieties, and from 0 to about 1 glyceryl moiety, and R₂ and R₃ contain from about 1 to about 20 3 carbon atoms and from 0 to about 1 hydroxy group, e.g., methyl, ethyl, propyl, hydroxyethyl, or hydroxypropyl radicals. The arrow in the formula is a conventional representation of a semipolar bond.

25 Examples of amine oxides suitable for use in this invention include dimethyldodecylamine oxide, oleyldi(2-hydroxyethyl) amine oxide, dimethyloctylamine oxide, dimethyl-decylamine oxide, dimethyl-tetradecylamine oxide, 3,6,9-trioxaheptacyldiethylamine oxide, di(2-hydroxyethyl)- 30 tetradecylamine oxide, 2-dodecoxyethyl dimethylamine oxide,

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3-dodecoxy-2-hydroxypropyl-di(3-hydroxypropyl)amine oxide,
diethylhexadecylamine oxide.

Non-limiting examples of preferred nonionic surfactants for
5 use herein are those selected from the group consisting of
C8-C14 glucose amides, C8-C14 alkyl polyglucosides, sucrose
cocoate, sucrose laurate, lauramine oxide, cocoamine oxide,
and mixtures thereof.

10 The term "amphoteric lathering surfactant," as used herein,
is also intended to encompass zwitterionic surfactants,
which are well known to formulators skilled in the art as a
subset of amphoteric surfactants.

15 A wide variety of amphoteric lathering surfactants can be
used in the compositions of the present invention.
Particularly useful are those which are broadly described as
derivatives of aliphatic secondary and tertiary amines,
preferably wherein the nitrogen is in a cationic state, in
20 which the aliphatic radicals can be straight or branched
chain and wherein one of the radicals contains an ionizable
water solubilizing group, e.g., carboxy, sulfonate, sulfate,
phosphate, or phosphonate.

25 Non-limiting examples of amphoteric surfactants useful in
the compositions of the present invention are disclosed in
McCutcheon's, Detergents and Emulsifiers, North American
Edition (1986), published by Allured Publishing Corporation;
and McCutcheon's, Functional Materials, North American
30 Edition (1992); both of which are incorporated by reference
herein in their entirety.

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Non-limiting examples of amphoteric or zwitterionic surfactants are those selected from the group consisting of betaines, sultaines, hydroxysultaines, alkyliminoacetates, iminodialkanoates, aminoalkanoates, and mixtures thereof.

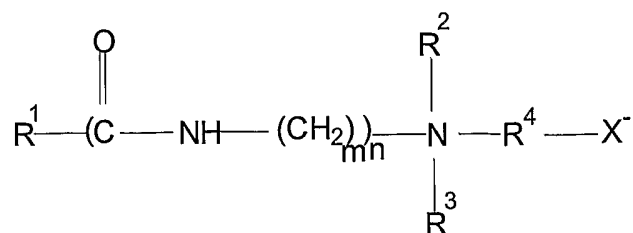
Examples of betaines include the higher alkyl betaines, such as coco dimethyl carboxymethyl betaine, lauryl dimethyl carboxymethyl betaine, lauryl dimethyl alphacarboxyethyl betaine, cetyl dimethyl carboxymethyl betaine, cetyl dimethyl betaine (available as Lonaine 16SP from Lonza Corp.), lauryl bis-(2-hydroxyethyl) carboxymethyl betaine, oleyl dimethyl gamma-carboxypropyl betaine, lauryl bis-(hydroxypropyl)alpha-carboxyethyl betaine, coco dimethyl sulfopropyl betaine, lauryl dimethyl sulfoethyl betaine, lauryl bis-(2-hydroxyethyl) sulfopropyl betaine, amidobetaines and amidosulfobetaines (wherein the $RCONH(CH_2)_3$ radical is attached to the nitrogen atom of the betaine), oleyl betaine (available as amphoteric Velvetex OLB-50 from Henkel), and cocamidopropyl betaine (available as Velvetex BK-35 and BA-35 from Henkel).

Example of sultaines and hydroxysultaines include materials such as cocamidopropyl hydroxysultaine (available as Mirataine CBS from Rhone-Poulenc).

Preferred for use herein are amphoteric surfactants having the following structure:

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- 15 -



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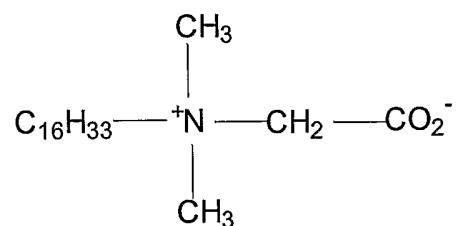
wherein R¹ is unsubstituted, saturated or unsaturated, straight or branched chain alkyl having from about 9 to about 22 carbon atoms. Preferred R¹ has from about 11 to about 18 carbon atoms; more preferably from about 12 to about 18 carbon atoms; more preferably still from about 14 to about 18 carbon atoms; m is an integer from 1 to about 3, more preferably from about 2 to about 3, and more preferably about 3; n is either 0 or 1, preferably 1; R² and R³ are independently selected from the group consisting of alkyl having from 1 to about 3 carbon atoms, unsubstituted or mono-substituted with hydroxy, preferred R² and R³ are CH₃; X is selected from the group consisting of CO₂, SO₃ and SO₄; R⁴ is selected from the group consisting of saturated or unsaturated, straight or branched chain alkyl, unsubstituted or mono-substituted with hydroxy, having from 1 to about 5 carbon atoms. When X is CO₂, R⁴ preferably has 1 to 3 carbon atoms, more preferably 1 carbon atom. When X is SO₃ or SO₄, R⁴ preferably has from about 2 to about 4 carbon atoms, more preferably 3 carbon atoms.

Examples of amphoteric surfactants of the present invention include the following compounds:

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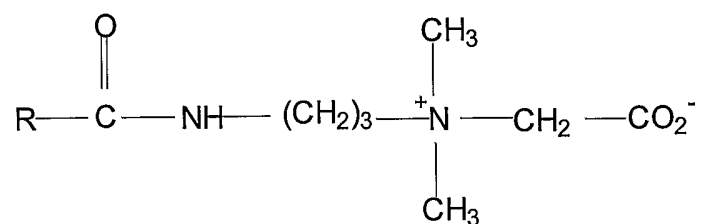
Cetyl dimethyl betaine (this material also has the CTFA designation cetyl betaine);

5



10 Cocamidopropylbetaine

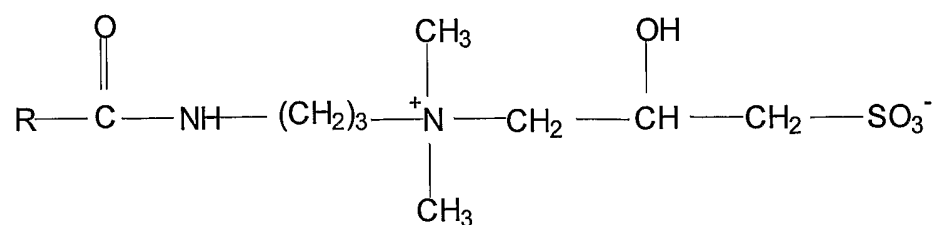
15



Cocamidopropyl hydroxy sultaine

20 wherein R has from about 9 to about 13 carbon atoms

25



30 wherein R has from about 9 to about 13 carbon atoms.

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Cationic surfactants are another useful class of surfactants that can be employed as auxiliary agents. They are particularly useful as additives to enhance skin feel, and provide skin conditioning benefits. One class of cationic surfactants is heterocyclic ammonium salts such as cetyl or stearyl pyridinium chloride, alkyl amidoethyl pyrrolidinium methyl sulfate, lapyrium chloride.

Tetra alkyl ammonium salts is another useful class of cationic surfactants. Examples include cetyl or stearyl trimethyl ammonium chloride or bromide; hydrogenated palm or tallow trimethylammonium halides; behenyl trimethyl ammonium halides or methyl sulfates; decyl isononyl dimethyl ammonium halides; ditallow (or distearyl) dimethyl ammonium halides; behenyl dimethyl ammonium chloride.

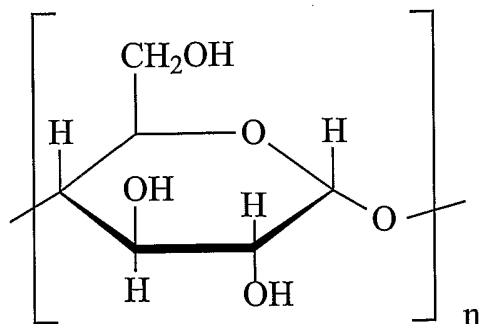
Other types of cationic surfactants that can be employed are the various ethoxylated quaternary amines and ester quats. Examples are PEG-5 stearyl ammonium lactate (e.g., Genamin KSL manufactured by Clarion), PEG-2 coco ammonium chloride, PEG-15 hydrogenated tallow ammonium chloride, PEG 15 stearyl ammonium chloride, dialmitoyl ethyl methyl ammonium chloride, dipalmitoyl hydroxyethyl methyl sulfate, stearyl amidopropyl dimethylamine lactate.

Still other useful cationic surfactants are quaternized hydrolysates of silk, wheat, and keratin proteins.

The compound which added to the formulation which induces formation of biphasic (multiphasic) liquid is polydextrose. Generally, the polydextrose has a formulation as follows:

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5



10 wherein n (defining number of linking glucose units) is from about 4 to about 22.

The biphasic inducing polydextrose compounds of the invention may also be defined by molecular weight in that
15 they should have MW range of from 600 to about 3600, more preferably 700 to 3000, more preferably 700 to 1800, more preferably 900 to 1500.

Whether defined by glucose units or MW, it should be
20 understood that the critical point is that the structure is such as to induce formation of a multiphasic/biphasic formulation defined by those characteristics which in turn define the biphasic liquid (e.g., viscosity of and stability in the biphasic state).

25

The amount of polydextrose used to induce biphasic state may vary depending on whether salt/electrolyte is used.

Thus, for example, if no salt is used (use of no or little
30 salt also distinguishes this invention from other biphasic liquids of the art where relatively large amounts of salt,

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e.g., greater than 3 % by wt., are in fact required to induce the biphasic liquid), then there is needed at least 15 % by wt. of polydextrose to induce biphasic separation. If some salt is added (e.g., at least 0.5 %, preferably at least 1.0 %), the amount of polydextrose needed goes down to 10 % by wt. If at least 2 % salt is used, the amount of polydextrose may be 5 %.

There is also generally a balance between amount of surfactant used, and amount of polydextrose. Generally lower surfactant requires more polydextrose and, conversely, more surfactant requires less polydextrose. Thus, for example, 5 % to 10 % by wt. surfactant may require about 40 % or more polydextrose, and 35 % surfactant may need only about 10-15 % polydextrose, even in the absence of salt.

Generally, the upper limit of polydextrose used is about 75 %. This is not an upper limit with regard to inducing biphasic liquid.

If electrolyte/salt is used, it typically will be used in amount of 0.5 % to no higher than 4 %, preferably no higher than about 3 % by wt. of total composition.

Preferably, the electrolyte is not a chelating electrolyte (these are generally poor in biodegradability).

Typically, the electrolyte should be a salt of a sulphate, bisulfate, carbonate, bicarbonate, phosphate, chloride, etc. Examples include sodium sulphate, potassium sulphate, ammonium sulphate, sodium chloride, and magnesium chloride.

- 20 -

Magnesium sulphate and sodium chloride are particularly preferred.

Finally, the balance of composition is water and minors.

5

The following optional ingredients may be used in the multiphasic/biphasic compositions of the invention.

The composition may contain polyalkylene glycol. The
10 polyalkylene glycol should be an alcohol, glycol or polyether of minimal molecular weight which is not irritating to the skin.

Examples of such include alcohols, particularly polyalkylene
15 oxides having MW 200-6000, preferably 200 to 3000. The polyalkylene glycol can be comprised of ethylene oxide, propylene oxide, butylene oxide or their mixtures either as polymers or copolymers. Specific examples include polyethylene glycols such as PEG 400. As noted, use of such
20 alcohols is not required.

The composition may further comprise thickeners. Generally, the thickener/viscosity modifier serves to thicken the upper and/or lower layer.

25

Thickeners which may be used include hydrophobically modified polyethers. Examples of this class of thickeners which may be used include but are not limited to sugar esters such as PEG (160) sorbitan triisostearate (Rheodol
30 TWS -399C ex. Kao Chemicals) or PEG-120 Pentaerythrityl Tetrastearate ex. Croda. Other examples include Glucam DOE

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120 (PEG 120 Methyl Glucose Dioleate); Rewoderm[®] (PEG modified glyceryl cocoate, palmate or tallowate) from Rewo Chemicals; Antil[®] 141 (from Goldschmidt); and Carbopol[®] polymers from Noveon.

5

Another class of suitable polymers are hydrophobically modified cellulose ethers including but not limited to hydroxyethyl cellulose, hydroxypropylcellulose and cellulose ethers with long pendant chains such as nonoxynyl hydroxyethylcellulose (Amerchol Polymer HM 1500).

10

Another class of suitable polymers are the hydrophobically modified acrylate copolymers such as Antil 208[®] (ex Goldschmidt) (acrylate/steareth-50 acrylate copolymer).

15

Another class of suitable polymers are the hydrophobically modified polyurethanes such as Acrysol series (e.g., Acrysol RM-2020) from Rhom and Haas.

20

Another class of suitable thickeners are xanthan gums, guar gums and chemically modified guar gums.

25

In addition to the ingredients noted above, the compositions of the invention may contain hydrotropes including but not limited to short chain monohydric or dihydric alcohols, xylene sulphonate and hexylene glycol. The purpose of these is to avoid the formation of liquid crystal phases resulting from the separation of the surfactant material into the upper phase, hence increasing its apparent concentration.

30

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The compositions may comprise benefit agents. Benefit agent may be any material that has potential to provide an effect on, for example, the skin.

5 The benefit agent may be water insoluble material that can protect, moisturize or condition the skin upon deposition from compositions of invention. These may include silicon oils and gums, fats and oils, waxes, hydrocarbons (e.g., petrolatum), higher fatty acids and esters, vitamins,
10 sunscreens. They may include any of the agents, for example, mentioned at column 8, line 31 to column 9, line 13 of U.S. Patent No. 5,759,969, hereby incorporated by reference into the subject application.

15 The benefit agent may also be a water-soluble material such as glycerin, polyols (e.g., saccharides), enzyme and α -Or β -hydroxy acid, either alone or entrapped in an oily benefit agent.

20 The benefit agent may be found in either the upper or the lower layer, depending on its solubility and partition coefficient; for example, oil may partition into the upper layer while more water soluble agents (e.g., α -hydroxyacids) may go into the lower.

25 The compositions may comprise perfumes, sequestering agents such as EDTA EHDP in amounts 0.01 % to 1 %, preferably 0.01 % to 0.05 %; coloring agents, opacifiers and pearlizers such as zinc stearate, magnesium stearate, TiO₂, mica, EGMS
30 (ethylene glycol monostearate) or styrene/acrylate copolymers.

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The compositions may further comprise antimicrobials such as 2-hydroxy 4,2'4' trichlorodiphenylether (DP300), 3,4,4'-trichlorocarbanilide, essential oils and preservatives such as dimethyl hydantoin (Glydant XL 1000), parabens, sorbic acid etc.

The compositions may also comprise coconut acyl mono or diethanol amides as suds boosters, and strongly ionizing salts such as sodium chloride and sodium sulfate may also be used to advantage.

Antioxidants such as, for example, butylated hydroxytoluene (BHT) may be used advantageously in amounts of about 0.01 % or higher if appropriate.

Cationic conditioners which may be used including Quatrisoft LM-200 Polyquaternium-24, Merquat Plus 3330- Polyquaternium 39; and Jaguar[®] type conditioners.

Composition may also include clays such as Bentonite[®] claims as well as particulates such as abrasives, glitter, and shimmer.

The compositions of the invention, when unmixed, typically have a viscosity of the lower layer which is lower than the viscosity of the upper layer, and a density of the lower layer which is greater than the density of the upper layer.

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The compositions of the invention, in a separated state, are also stable in that no recrystallization (e.g., in the lower layer) occurs even when left sitting for more than 6 months at temperature of 0°C.

5

Compositions of the invention have an experiential element in that they are intended to be agitated by the consumer to mix and form a single visible phase before separating again after a time, which can be anywhere from about a few seconds
10 to not more than about 24 hours.

When mixed, the compositions typically have a viscosity in the range of 100 to 5000, preferably 200-400 mPas at a shear rate of 10s^{-1} at 25°C at a shear rate of 10s^{-1} at 25°C, as
15 measured by using Haake RV20 Rotivisco Rheometer.

Finally, the packages in which the compositions are contained are preferably translucent or transparent. By this is meant that the materials (e.g., plastics) have a
20 light transmittance of greater than 50 %, preferably greater than 75 %, more preferably greater than 85 % as measured at wavelength of 460 nm as determined by standard spectroscopy method. In practical terms the package should preferably be sufficiently transparent to permit the separation of the two
25 or more layers to be visible to the naked eye.

Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts or ratios of materials or
30 conditions or reaction, physical properties of materials

- 25 -

and/or use are to be understood as modified by the word "about".

Where used in the specification, the term "comprising" is
5 intended to include the presence of stated features, integers, steps, components, but not to preclude the presence or addition of one or more features, integers, steps, components or groups thereof.

10 The following examples are intended to further illustrate the invention and are not intended to limit the invention in any way.

Unless indicated otherwise, all percentages are intended to
15 be percentages by weight.

Methodology

Measurement of Viscosity

20

Description

A Haake Rheometer was used to measure the viscosities of liquid and semi-solid personal wash products in the small
25 scale with the viscosity measured at various shear rates.

Equipment

The instrument was an RV 20 Rotovisco RC 20 rheometer which
30 includes a stand and sample temperature control unit, cups and bobs for loading the sample, a waterbath which is

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maintained at 25°C and a computer and plotter to manipulate and record the data.

Operational Procedure

5

The Haake rheometer, computer, monitor and printer were turned on.

10 Water bath: The water bath was filled with water to a required level, the appropriate temperature was set and water bath was turned on.

Measurement Systems: The sample was loaded into rheometer and equilibrated to 25°C.

- 15 a) the appropriate cup and bob for the product are selected as noted below.
- i) NV for viscosity measurements of low viscous products, e.g. diluted solutions, fruit juices, etc;
 - 20 ii) SV1 for viscosity measurements of high viscosity liquids working in the low to medium range which consists of a SV cup with a rotor(bob). This is the cup and bob that is typically used to measure shower gel
 - 25 products;
- b) the rotor(bob) was secured on to the top segment of the measuring system;
- c) the RV 20 rheometer was adjusted using the zero button;
- 30 d) the sample was poured into the cup until almost three quarters filled (approx. 20 g), and then the

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cup was carefully slid through the temperature controller and screwed to the main segment of the rheometer so that it was immersed in the product and sample was slightly above the rim of the bob;

- 5 e) waited 5 to 10 minutes after loading the sample to ensure equilibration of sample to set temperature (set parameters on computer while waiting for temperature equilibration).

10 Computer

- a) a floppy disc was inserted and previous standard file was loaded if one is already saved on disc. If not, the following details were loaded into the computer:

- 15 i) measurement: select SV1, NV1, SV2P depending on the spindle used;
ii) select four segments for four shear rates, 1, 10, 100, 400 at 25°C and in 10 steps;
- b) on the computer screen follow the steps below to load the above details:
20 measurement - identification (record details of the sample);
measurement - parameter - select SV1;
measurement - go immediately (after sample is
25 equilibrated);
- c) this starts the measurement which takes about 10 minutes;
- d) once the measurement was completed, results were saved on floppy disk; results were either printed
30 or set as graphical representation.

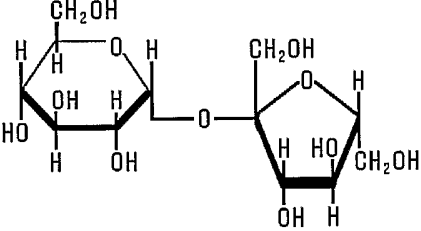
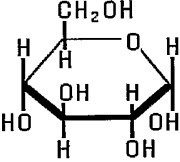
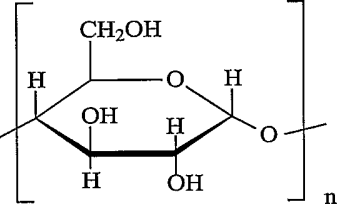
Results

The results were recorded as viscosity in mPas (cps) at the shear rates: 1/sec, 10/sec and 100/sec. The temperature and
5 spindle (bob) size were recorded with each sample measurement.

Materials & Methods

10 Materials

Table 2: Raw Materials

	Trade Name	Structure
Na-Laureth Ether Sulfate	Steol CS-230	--
Coco Amido Propyl Betaine	Tegobetaine F-50	--
Almeo Blend	Almeo Blend	--
Sorbitol	Sorbitol	$ \begin{array}{c} \text{CH}_2\text{OH} \\ \\ \text{HCOH} \\ \\ \text{HOCH} \\ \\ \text{HCOH} \\ \\ \text{HCOH} \\ \\ \text{CH}_2\text{OH} \end{array} $
Sucrose	Sucrose	
Glucose	Glucose	
Polydextrose (Av. MW= 3600)	Maltrine M40	
Polydextrose (Av. MW= 1800)	Maltrine M100	
Polydextrose (Av. MW= 1000)	Maltrine M180	
Polydextrose (Av. MW= 720)	Maltrine M250	
Magnesium Sulfate	Mg*SO4	--

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Formulation Preparation

A simple surfactant solution was prepared at about 5 wt.% to about 35.0 wt.% without any saccharides. Then saccharides were added to desired level. After adding saccharides, sample was heated for 1 hour at 60°C to dissolve any solid materials, then allowed to cool to room temperature. Once sample reached equilibrium at room temperature, it was mixed by shaking and observation are made.

10

Viscosity & Product Appearance

Formulations were screened for viscosity using standard PW protocols as set forth in methodology section above. The formulations were observed for any discoloration and re-crystallisation of saccharides at room temperature.

15

Examples

20 Examples 1-6 and Comparative 1-4

Polydextrose (i.e., Polydextrose M180) was examined for its ability to promote the formation of biphasic shower gel formulations, compared to sucrose, sorbitol and glucose.

25 Results are set forth in Table 1 and 2 below.

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Table 1: Sucrose, Sorbitol, Glucose and Polydextrose
Comparison

	Comparative 1	Comparative 2	Comparative 3	Example 1	Example 2
Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients
Na-Laureth Ether Sulfate	10.0	10.0	10.0	10.0	8.3
CocoAmido Propyl Betaine	5.0	5.0	5.0	5.0	8.3
Sucrose	10-50	--	--	--	--
Sorbitol	--	10-50	--	--	--
Glucose	--	--	10-50	--	--
Polydextrose M180 (avg MW=1000)	--	--	--	40.0	33.3
MgSO ₄	--	--	--	--	--
NaCl	--	--	--	--	--
PEG-400	--	--	--	--	--
Water	Q.S to 100	Q.S to 100	Q.S to 100	Q.S to 100	Q.S to 100
Biphasic	NO	NO	NO	Yes, Slight	Yes, 80:20

5 As seen from Table 1, when 15-16% surfactant is used
(SLES/CAPB), only the polydextrose was able to promote phase
separation in the absence of salt (Example 1 and 2)

In Table 2, shown below, the applicants test for the effect
10 of salt (e.g., MgSO₄) as well as for the effect of
surfactant level versus amount of polydextrose needed.

Table 2

	Comparative 4	Example 3	Example 4	Example 5	Example 6
Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients
Na-Laureth Ether Sulfate	8.3-11.5	8.3-11.5	8.3	3.3	23.3
CocoAmido Propyl Betaine	8.3-11.5	8.3-11.5	8.2	1.7	11.7
Polydextrose M040 (avg MW=3600)	--	--	--	--	--
Polydextrose M100 (avg MW=1800)	23.0-35.0	--	--	--	--
Polydextrose M180 (avg MW=1000)		23.0-35.0	20.0	60.0	15.0
Polydextrose M250 9avg MW=720)	--	--	--	--	--
MgSO ₄	--	--	1.0-3.0	--	--
NaCl	--	--	--	--	--
PEG-400	--	--	--	--	--
Water	Q.S to 100	Q.S to 100	Q.S to 100	Q.S to 100	Q.S to 100
Biphasic	NO	Yes, 60:40	Yes, 80:20 to 50:50	Yes, 90:10	Yes, 90:10

5 This table also shows various points. First, the table shows that the polydextrose level can be lowered without increasing surfactant level, if small amounts of salt (e.g., MgSO₄) are used (see Example 4). Also, Table 2 shows that higher levels of polydextrose permit much lower levels of

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surfactant (Example 5) and, conversely, high levels of surfactant permit use of lower levels of polydextrose (Example 6). One other interesting point is that polydextrose M100 does not appear to form biphasic.

5 However, as seen in Table 3 below, when salt is added, biphasic is formed, even at lower surfactant levels.

Examples 7-12 and Comparative 5

10 Different surfactant systems are also able to produce biphasic formulations when combined with the proper levels of polydextrose and salt. As with SLES/CAPB, the blend of ammonium lauryl ether sulfate, ammonium lauryl sulfate and cocoylmonoethanolamide will also promote phase separation
15 (See Table 3 below). Examples 7-9 and Comparative 5 compare polydextrose M180 with and without salt in the surfactant mix. No phase separation is achieved with 25 % polydextrose M180 alone (Comparative 5), but phase separation can be achieved with incorporation of low levels of $MgSO_4$ or NaCl
20 (Formulations 7-9).

Also, the addition of low levels of salt promote phase separation with other polydextrose. Similar biphasic formulations can be produced with polydextrose M250 (Example
25 10), polydextrose M100 (Example 11) and polydextrose M040, although the lower layer is turbid in these three formulations (Table 3 below). By using low levels of salt, a number of different polydextrose materials with different molecular weights and varying numbers of glucose units can
30 be used to promote the formation of biphasic surfactant formulations.

Table 3: Surfactant Blend

	Comparative 5	Example 7	Example 8	Example 9	Example 10	Example 11	Example 12
Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients	% Ingredients
Na-Laureth Ether Sulfate	--	--	--	--	--	--	--
CocoAmido Propyl Betaine	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Ammonium Lauryl Sulfate; Ammonium Laurylether Sulfate; Cocomoethanolamide	13.0	13.0	13.0	13.0	13.0	10.0	10.0
Polydextrose M040 (avg. MW=3600)	--	--	--	--	--	--	5.0
Polydextrose M100 (avg MW=1800)	--	--	--	--	--	5.0	--
Polydextrose M180 (avg MW=1000)	25.0	20.0	20.0	20.0	--	--	--
Polydextrose M250 (avg MW=720)	--	--	--	--	20.0	--	--
MgSO4	0.0	1.0	1.0	--	--	--	--
NaCl	--	--	--	1.5	3.0	3.0	3.0
PEG-400	--	--	2.0	--	--	--	--
Water	Q.S to 100	Q.S to 100	Q.S to 100	Q.S to 100	Q.S. to 100	Q.S. to 100	Q.S. to 100
Biphasic	NO	Yes, 60:40	Yes, 60:40	Yes, 80:20	Yes, 50:50	Yes, 80:20	Yes, 40:60

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Examples 13 and 14 below are similar to Examples 11 and 12 except they use 5 % salt instead of 3 %.

	Example 13	Example 14
Ingredients	% Ingredients	% Ingredients
Na-Laureth Ether Sulfate	--	--
CocoAmido Propyl Betaine	2.0	2.0
Ammonium Lauryl Sulfate; Ammonium Laurylether Sulfate; Cocomonoeethanolamide	10.0	10.0
Sucrose	--	--
Sorbitol	--	--
Glucose	--	--
Polydextrose M040 (avg. MW=3600)	--	5.0
Polydextrose M100 (avg MW=1800)	5.0	--
Polydextrose M180 (avg MW=1000)	--	--
Polydextrose M250 (avg MW=720)	--	--
MgSO ₄	--	--
NaCl	5.0	5.0
PEG-400	--	--
Water	Q.S to 100	Q.S to 100
Biphasic	Yes, 50:50	Yes, 70:30

5 Example 15

Table 4 below shows a variety of formulation parameters that lead to biphasic product formulations as indicated in the Table below.

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Table 4: Formulations parameters that lead to Biphasic product formations as indicated in the phase separation column.

% Total Surfactant	%MD 180	% MgSO4	%Water	Phase Separation	Top (cm)	Bottom (cm)	% Top Layer
15.0	10.0	1.00	74.0	NO	--	--	
14.3	14.3	0.95	70.45	NO	--	--	
13.6	18.2	0.91	67.29	NO	--	--	
13.0	21.7	0.87	64.43	NO	--	--	
20.0	10.0	1.00	69.0	NO	--	--	
19.0	14.3	0.95	65.75	NO	--	--	
18.2	18.2	0.91	62.69	NO	--	--	
17.4	21.7	0.87	60.03	NO	--	--	
25.0	10.0	1.00	64.0	NO	--	--	
23.8	14.3	0.95	60.95	YES	3.5	0.5	87.5
22.7	18.2	0.91	58.19	YES	3.5	0.5	87.5
21.7	21.7	0.87	55.73	YES	3.4	0.6	85.0
15.0	10.0	2.00	73.0	NO	--	--	
14.3	14.3	1.90	69.5	NO	--	--	
13.6	18.2	1.82	66.38	YES	3.4	0.6	85.0
13.0	21.7	1.74	63.56	YES	2.1	1.9	52.5
20.0	10.0	2.00	68.0	NO	--	--	
19.0	14.3	1.90	64.8	YES	3.0	1.0	75.0
18.2	18.2	1.82	61.78	YES	2.8	1.2	70.0
17.4	21.7	1.74	59.16	YES	2.7	1.3	67.5
25.0	10.0	2.00	63.0	YES	3.7	0.3	92.5
23.8	14.3	1.90	60.0	NO	--	--	
22.7	18.2	1.82	57.28	YES	3.6	0.4	90.0
21.7	21.7	1.74	54.86	YES	3.0	1.0	75.0
15.0	10.0	3.00	72.0	YES	3.5	0.5	87.5
14.3	14.3	2.86	68.54	YES	2.8	1.2	70.0
13.6	18.2	2.73	65.47	YES	2.2	1.8	55.0

Table 4 (Continued)

% Total Surfactant	%MD 180	% MgSO ₄	%Water	Phase Separation	Top (cm)	Bottom (cm)	% Top Layer
13.0	21.7	2.61	62.69	YES	1.8	2.2	45.0
20.0	10	3.00	67.0	YES	3.5	0.5	87.5
19.0	14.29	2.86	63.85	YES	2.9	1.1	72.5
18.2	18.18	2.73	60.89	YES	3.3	0.7	82.5
17.4	21.74	2.61	58.25	YES	2.2	1.8	55.0
25.0	10	3.00	62.0	NO	--	--	
23.8	14.29	2.86	59.05	NO	--	--	
22.7	18.18	2.73	56.39	YES	2.2	1.4	61.1
21.7	21.74	2.61	53.95	YES	2.8	1.2	70.0
15.0	0.00	0	85.0	NO	--	--	
13.6	9.09	0	77.31	NO	--	--	
13.0	13.04	0	73.96	NO	--	--	
12.5	16.67	0	70.83	NO	--	--	
12.0	20.00	0	68.0	NO	--	--	
10.7	28.57	0	60.73	NO	--	--	
20.0	0.00	0	80.0	NO	--	--	
18.2	9.09	0	72.71	NO	--	--	
17.4	13.04	0	69.56	NO	--	--	
16.7	16.67	0	66.63	NO	--	--	
16.0	20.00	0	64.0	YES	3.5	0.5	87.5
14.3	28.57	0	57.13	YES	3.1	0.9	77.5
13.8	31.03	0	55.17	YES	3.1	0.9	77.5
13.3	33.33	0	53.4	YES	3.1	0.9	77.5
12.5	37.50	0	50.0	YES	3.0	1.0	75.0
11.8	41.18	0	47.02	NO	--	--	
25.0	0.00	0	75.0	NO	--	--	
22.7	9.09	0	68.21	NO	--	--	
21.7	13.04	0	65.26	NO	--	--	
20.8	16.67	0	62.53	NO	--	--	
20.0	20.00	0	60.0	YES	3.6	0.4	90.0
19.2	23.08	0	57.72	YES	3.2	0.8	80.0
30.0	0.00	0	70.0	NO	--	--	
27.3	9.09	0	63.61	NO	--	--	
26.1	13.04	0	60.86	NO	--	--	
25.0	16.67	0	58.33	NO	--	--	
24.0	20.00	0	56.0	YES	3.2	0.8	80.0

Table 4 (Continued)

% Total Surfactant	%MD 180	% MgSO4	%Water	Phase Separation	Top (cm)	Bottom (cm)	% Top Layer
23.1	23.08	0	53.82	YES	3.2	0.8	80.0
22.2	25.93	0	51.87	YES	3.0	1.0	75.0
21.4	28.57	0	50.03	YES	3.0	1.0	75.0
20.0	33.33	0	46.67	YES	3.0	1.0	75.0
18.8	37.50	0	43.7	YES	2.8	1.2	70.0
17.6	41.18	0	41.22	YES	2.8	1.2	70.0
10.0	30.00	0	60.0	NO	--	--	
9.5	33.33	0	57.17	NO	--	--	
20.0	30.00	0	50.0	YES	3.0	1.0	75.0
19.0	33.33	0	47.67	YES	3.0	1.0	75.0
18.2	36.36	0	45.44	YES	2.8	1.2	70.0
17.4	39.13	0	43.47	YES	2.8	1.2	70.0
16.7	41.67	0	41.63	YES	2.4	1.6	60.0
15.4	46.15	0	38.45	YES	2.4	1.6	60.0
14.3	50.00	0	35.7	YES	2.5	1.5	62.5
30.0	20.00	0	50.0	YES	3.0	1.0	75.0
28.6	23.81	0	47.59	YES	3.0	1.0	75.0
27.3	27.27	0	45.43	YES	2.8	1.2	70.0
26.1	30.43	0	43.47	YES	2.8	1.2	70.0

CLAIMS

1. A liquid cleansing composition comprising:
 - 5 (a) about 5 % to 75 % by wt. of a surfactant selected from anionic surfactants, nonionic surfactants, amphoteric/zwitterionic surfactants, cationic surfactants and mixtures thereof;
 - 10 (b) at least about 15 % of a polydextrose molecule or molecules, wherein the degree of polymerization is about 4 to about 22 (corresponding to MW of about 600 to about 3600); and
 - (c) the balance water and minors;wherein the composition comprises at least two visibly separated aqueous based layers when left sitting without shaking or stirring.
15
2. A composition according to claim 1, comprising 6 % to 40 % surfactant.
- 20 3. A composition according to claim 1 or claim 2, wherein the MW of polydextrose is 700 to 1800.
4. A composition according to claim 3, wherein the MW is 900 to 1500.
25
5. A composition according to claim 4, wherein the MW is 900 to 1200.
6. A composition according to any of the preceding claims, comprising about 5 % to 10 % surfactant and greater than
30 about 40 % polydextrose.

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7. A composition according to any of the preceding claims, having greater than about 35 % surfactant and about 15 % and higher polydextrose.
- 5 8. A liquid cleansing composition comprising:
- (a) about 5 % to 75 % by wt. of a surfactant selected from anionic surfactants, nonionic surfactants, amphoteric/zwitterionic surfactants, cationic surfactants and mixtures thereof;
 - 10 (b) at least about 10 % of a polydextrose molecule or molecules, wherein the degree of polymerization is about 4 to about 22 (corresponding to MW of about 600 to about 3600); and
 - (c) at least about 0.5 % to about 3 % salt;
 - 15 (d) the balance water and minors;
- wherein the composition comprises at least two visibly separated aqueous based layers when left sitting without shaking or stirring.
- 20 9. A composition according to claim 8, comprising at least about 1 % to about 3 % salt.
10. A liquid cleansing composition comprising:
- 25 (a) about 5 % to 75 % by wt. of a surfactant selected from anionic surfactants, nonionic surfactants, amphoteric/zwitterionic surfactants, cationic surfactants and mixtures thereof;
 - (b) at least about 5 % of a polydextrose molecule or molecules, wherein the degree of polymerization is
 - 30 about 4 to about 22 (corresponding to MW of about 600 to about 3600); and

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(c) at least about 2 % salt;

(d) the balance water and minors;

wherein the composition comprises at least two visibly
separated aqueous based layers when left sitting without
shaking or stirring.

5

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 03/08590

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C11D3/22 C11D17/00 C11D1/94 C11D3/02 A61K7/00
A61K7/50 //C11D1:29,C11D1:14,C11D1:52,C11D1:90

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C11D A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 00 61716 A (HENKEL KGAA) 19 October 2000 (2000-10-19) page 2, paragraph 2 page 3, last paragraph -page 4, paragraph 2 page 5, paragraph 3 ---	1-10
A	US 6 429 177 B1 (SALMON TOM ET AL) 6 August 2002 (2002-08-06) cited in the application claims 1-7; examples ---	1-10
A	US 3 354 091 A (MACLEAN ALLISTER N ET AL) 21 November 1967 (1967-11-21) column 1, line 19-53 ---	1-10
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

° Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
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- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

28 November 2003

Date of mailing of the international search report

10/12/2003

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PCT/EP 03/08590

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 01 21753 A (HENKEL KGAA) 29 March 2001 (2001-03-29) page 3, paragraph 2 page 15, paragraph 1 page 23, last paragraph -----	1-10
A	WO 00 71665 A (HENKEL KGAA) 30 November 2000 (2000-11-30) page 2, line 22-27 page 6, line 17-28 page 12, line 31 -page 13, line 13; examples -----	1-10

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