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(54) Liquid detergent compositions.

(57) Aqueous surfactant concentrates comprise water and about 30 to 80% by weight of a surfactant mixture of (a) at least 50% by weight anionic surfactants based on the weight of the surfactant mixture, including at least 20% alkyl benzene sulphonate based on the weight of the surfactant mixture, and (b) at least 2%, based on the weight of the surfactant mixture, of nonionic ethoxylate having an HLB less than 12, the water being present in an amount adapted to form a spherulitic or lamellar composition. Liquid detergents contain water, from 5 to 25% by weight of said surfactant mixture and enough dissolved electrolyte to form a non-sedimenting spherulitic composition.

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LIQUID DETERGENT COMPOSITIONS

This invention relates to liquid detergent compositions and preferably compositions having a stable spherulitic structure and comprising a substantial proportion of non-ionic surfactant. The invention is especially applicable to the formulation of liquid detergent compositions having a controlled foaming character e.g. suitable for use in a front loading washing machine. It is also of value in preparing compositions suitable for use in low temperature wash regimes.

In formulating liquid laundry detergents, a good balance of mobility and high pay load of useful ingredients is most readily obtained if the surfactants can be constrained to adopt a spherulitic structure by the presence of an appropriate concentration of an electrolyte which salts surfactants out of aqueous micellar solution and forms them into spherulites, which are believed to consist of a number of concentric shells, each comprising a curved surfactant bilayer, alternating with shells of aqueous electrolyte solution. When the spherulites are sufficiently closely packed, a stable structured liquid is formed, capable of suspending solid particles of builder. Compositions of this type are discussed in detail in our GB-A-2 153 380, the disclosure of which is incorporated herein by reference.

Relatively few single surfactants are known which are capable of forming stable spherulitic compositions on their own. They are characterised by very bulky hydrophobic groups having a large cross-sectional area relative to the hydrophilic groups, e.g. some secondary or branched chain alkyl benzene sulphonates such as sodium dodeca-6-yl benzene sulphonate, dialkyl sulphosuccinates and some olefin sulphonates. Generally these surfactants are expensive and exhibit poor detergency. However spherulites can more readily be obtained using mixtures of surfactants. In particular mixtures of linear alkyl benzene sulphonates with alkyl ether sulphates, amineoxides, or alkanolamides are all capable of forming stable spherulitic compositions and exhibit relatively cost-effective detergency, at least for natural fibres.

Such compositions however have two disadvantages, which apply, respectively to those markets where front loading washing machines are commonly used, and to markets in which washing is carried out at low or ambient temperatures and/or the washload includes a high proportion of synthetic fibre.

Firstly the surfactant mixtures hitherto found to give stable spherulites have been inherently high foaming systems. To formulate detergent compositions suitable for use in front loading washing machines, it has therefore been necessary to include relatively large amounts of expensive antifoams such as silicones. In addition to the cost, silicones have a less than ideal performance in that they tend to give too low a foam in the wash but some residual foam in the rinse. The consumer gets the impression that the detergent is not very effective and is not rinsing out properly.

Secondly it has not proved possible to make stable spherulitic compositions containing significant levels of ethoxylated non-ionic detergent surfactants. Mixtures containing the latter are generally more cost-effective for cleaning synthetic fibres and give improved performance at low wash temperatures e.g. below 40°C. The detergent surfactants are generally defined as those having an HLB between 12 and 15. Surfactants outside this HLB range are not normally considered to be detergents.

A further problem which may arise when liquid detergents are exported or sold at a point remote from the source of surfactant is the cost of transporting the water and builder content of the liquid detergent. It is convenient to be able to transport the active material as a concentrate in a form suitable for blending and dilution at a site closer to the point of sale. Such compositions need to be pourable and non-separating, but also to be relatively the cost of transporting the water and builder content of the liquid detergent. It is convenient to be able to transport the active material as a concentrate in a form suitable for blending and dilution at a site closer to the point of sale. Such compositions need to be pourable and non-separating, but also to be relatively concentrated. Many bulk surfactants, however, are difficult to concentrate above about 30% due to the formation of immobile M phases.

We have now discovered that certain mixtures of linear alkyl benzene sulphonates with non-ionic surfactants having an HLB of less than 12 form stable spherulitic compositions with high levels of wash performance on both natural and synthetic fibres and have substantially reduced foaming tendencies, and/or improved wash performance at low temperatures.

We have further discovered that when such mixtures are concentrated to appropriate concentrations within the range 25 to 80%, non-separating pourable spherulitic and/or G phase compositions are obtained which can readily be diluted or blended with other detergent ingredients and which are relatively cheap to transport and store.

Our invention, according to one embodiment, provides a surfactant system for use in a liquid detergent consisting essentially of: an active mixture of (a) at least 50% by weight, based on the total weight of the surfactants present, of anionic surfactants comprising at least 20%, based on the total weight of the

surfactants present, of an alkyl benzene sulphonate, and (b) at least 2% by weight, based on the total weight of the surfactants present, of an alkoxylated non-ionic surfactant having an HLB below 12; and sufficient water to provide a non-separating pourable spherulitic or G phase composition.

Preferably the surfactant concentration of the surfactant system is greater than 28%, e.g. greater than 30% and preferably between 30 and 80%. Usually the surfactant concentration is greater than 35% e.g. 40 to 75% especially 50 to 70% by weight.

The surfactant system preferably consists essentially of surfactant and water together with such traces of sodium sulphate as are unavoidably present as an impurity in the anionic surfactant. It is, however, possible to include minor proportions of non-surfactant electrolytes in order to modify the viscosity and/or phase structure of the composition. We generally prefer that such electrolytes be present in proportions of less than 10%, preferably less than 5%, usually less than 2%, e.g. less than 1% of the total weight of the surfactant system. The presence of organic solvents is not preferred.

According to a further embodiment our invention provides a liquid detergent composition comprising water, sufficient of an active mixture as aforesaid to provide a total surfactant concentration of from 5 to 25%, by weight of the composition; and a dissolved surfactant desolubilising electrolyte in a concentration adapted to provide a non-sedimenting spherulitic composition. Preferably the liquid detergent composition of our invention additionally comprises suspended solid particles of a builder.

The alkyl benzene sulphonate preferably has an average of from 10 to 14 aliphatic carbon atoms, e.g. 12, and is preferably a linear alkyl benzene sulphonate. Particularly preferred are alkyl benzene sulphonates containing a broad range of different homologues and isomers.

For low foam use we prefer that the anionic surfactant comprises at least one C14-22 mono-and/or di-alkyl phosphate ester in a proportion of up to 50% e.g. 2 to 40%, preferably 5 to 30% especially 10 to 20% of the total weight of the anionic surfactant present.

The anionic surfactant may additionally or alternatively comprise other sulphonated and/or sulphated surfactants and/or carboxylates. For instance the anionic surfactant may comprise a mixture of alkyl benzene sulphonate with from 5 to 100%, preferably 10 to 50% e.g. 15 to 30% of a C_{8-20} (preferably C_{10-14}) alkyl sulphate, based on the weight of alkyl benzene sulphonate. Where low temperature performance is important and low foam is not required the composition preferably comprises from 5 to 150%, preferably 20 to 120% e.g. 50 to 110% of C_{8-20} (preferably C_{16-18}) alkyl polyalkyleneoxy sulphate based on the weight of alkylbezene sulphonate. The alkyl polyalkyleneoxy sulphate preferably comprises an average of from 1 to 10 e.g. 1.5 to 5 molar parts of ethylene oxide. Other anionic surfactants which may be present include olefin sulphonates, parafin sulphonates, fatty acid sulphonates, sulphosuccinates, sulphosuccinamates and soaps. The counter ion of the anionic surfactant may be sodium, lithium, potassium, ammonium, or a lower alkyl ammonium or mono-di- or tri-alkanolammonium or a mixture of such ions.

The non-ionic surfactant is preferably an ethoxylated alcohol but may alternatively be an ethoxylated carboxylic acid, alkyl amine, alkanolamide, alkylphenol, or ester such as an ethoxylated glyceryl, sorbitan, phosphate or phosphonate ester.

The alkyl groups of the non-ionic surfactant may be straight chain and primary, but comprise preferably at least some branched chain alkyl groups. It is often found that the greater the proportion of branched chain alkyl groups and the amount of branching, the more effective the detergency of the product.

The alkyl groups of the non-ionic surfactant have an average of from 8 to 20, preferably 10 to 18 carbon atoms. For example ethoxylated C_{9-15} e.g. C_{9-11} or C_{12-13} branched chain alcohols are especially preferred from the point of view of detergency, but ethoxylated C_{16-18} linear alcohols give suerior foam inhibition.

For the lower temperature wash conditions, C_{8-15} alkyl groups are preferred.

The number of ethylene oxide groups is selected, according to the alkyl chain length, to provide the desired HLB. Typically there are from 1 to 12, e.g. 2 to 8, ethylene oxide groups. It is possible to replace one or more of the ethylene oxide groups with propylene oxide. The HLB of the non-ionic surfactant is preferably from 2 to 12 e.g. 3 to 10 especially 3.5 to 8.

We prefer ethoxylates of feedstocks containing at least 20%, more preferably at least 30%, especially at least 50%, e.g. at least 60%, branched chain alcohols.

The non-ionic surfactant may comprise a proportion of free C_{8-22} alcohol. This is especially preferred for low foam compositions. The latter typically contain C14 to 22 fatty alcohols, e.g. in proportions up to 100% by weight of the total anionic surfactant. High foam, low temperature compositions may contain minor proportions of unethoxylated fatty alcohols, corresponding to the feed stock of the fatty alcohol ethoxylate. The latter are usually C_9 to $_5$, e.g. C_{9-11} alcohols. We do not exclude the presence of polyethylene glycol in the nonionic surfactant.

Any fatty alcohol or polyethylene glycol are deemed to be included in the total ethoxylated non-ionic

surfactant when estimating the HLB of the latter or in defining relative proportions of non-ionic surfactant. In the case of ethoxylated fatty alcohols the HLB may be taken for the purpose of this specification as being one fifth of the weight percentage of ethylene oxide based on the weight of non-ionic surfactant.

The total proportion of surfactant is from 5 to 25% of the liquid of the total weight detergent composition. For low foam applications the total proportion of surfactant is preferably less than 20%, e.g. less than 14%, most preferably 6 to 12% based on the weight of the liquid detergent composition. However for low temperature use, where low foam is not a requirement, higher concentrations of total surfactant are preferred e.g. greater than 18%, especially 20 to 25% based on the total weight of the liquid detergent composition.

The detergent composition of our invention comprises an electrolyte in a quantity sufficient to provide a non-sedimenting spherulitic composition. "Electrolyte" is used herein to refer to any water-soluble compound which is dissolved in the composition, which ionises in solution and which is capable of salting the surfactants out of micellar solution to form a spherulitic composition, but which is not itself a surfactant. The electrolyte may, preferably, comprise any of the water-soluble builders, or a dissolved portion of a sparingly soluble builder. Examples of suitable electrolytes therefore include citrates, orthophosphates, nitrilotriacetates, condensed phosphates such as pyrophosphates, tripolyphosphates or tetraphosphates, soluble phosphate glasses such as hexametaphosphates, carbonates, silicates, phosphonates, such as acetodiphosphonates, aminotris(methylene phosphonates), ethylene diamine tetrakis(methylene phosphonates), diethylene triamine pentakis(methylene phosphonates) and higher homologues of the same series, and ethylenediamine tetracetates.

It is possible to use as electrolyte, or part thereof, a buffer such as a borate, or less preferably a non-functional salt such as a chloride, bromide, iodide, nitrate or the like. Organic electrolytes such as formate or acetate may also be present.

Sulphate is normally present in minor amounts as an impurity of the anionic surfactant, but may give rise to undesirable crystallisation in amounts greater than about 3%. We therefore prefer to avoid the use of higher concentrations of sulphate.

The cationic portion of the electrolyte is preferably an alkali metal, e.g. lithium, potassium or, more preferably, sodium.

The total concentration of dissolved electrolyte is preferably greater than 1%, typically 2 to 12%, depending upon the concentration of surfactant and the total proportion of non-ionic to anionic. Higher concentrations of surfactant and/or higher proportions of non-ionic surfactant require lower concentrations of electrolyte to form non-separating, spherulitic compositions. If the concentration of surfactant and/or the proportion of non-ionic surfactant is sufficiently high, any adventitious sodium sulphate may be sufficient to ensure a stable spherulitic composition without the adition of extra electrolyte.

The builder is preferably sodium tripolyphosphate and is preferably present in excess of its solubility, as suspended solid particles. Alternatively or additionally the composition may contain zeolites and/or solid particles of any of the builders referred to above under the discussion of electrolytes.

The low-foaming compositions of our invention containing phosphate esters have been found to prevent excessive foaming but not to suppress foaming entirely at the wash stage, giving visible confirmation that the detergent is present and acting. There is however little or no foaming in the rinse water.

It is possible, but less preferred, to add other foam suppressing agents such as C_{16-18} fatty alkanolamides and/or hydrocarbons in addition to or instead of the phosphate ester. We prefer, however, not to use silicones as the antifoams, although it is possible, according to our invention, to achieve effective foam suppression with reduced proportions of silicone antifoam compared with those conventionally used. Minor amounts of silicone may be added as protective agent if it is desired to include enzymes in the formulation, for example as described in European Patent Application 87301489.2.

Detergent compositions of our invention may contain the usual minor ingredients. Preferably these include an anti-redeposition agent such as sodium carboxymethyl cellulose, in conventional amounts, an optical brightener and perfumes and/or colouring. The compositions may contain suspended particles of fabric softeners such as bentonite or synthetic clays, or may generally contain any of the other minor ingredients, builders and electrolytes listed in our aforesaid GB-A-2153380 and in substantially the same proportions. A specially preferred liquid detergent composition for low foam use comprises.. water; from 4 to 10%, preferably 5 to 8% of C_{12-14} linear alkyl benzene sulphonate; from 0.5 to 4%, preferably 1 to 3% of C_{10-14} alkyl sulphate; from 0.5 to 4%, preferably 1 to 3% of an ethoxylated alcohol said alcohol having from 10 to 14, preferably 11 to 13 carbon atoms and said ethoxylated alcohol having an average of from 1 to 5, preferably from 2 to 4 ethylenoxy group per molecule; and from 20 to 30% e.g. 24 to 27% of sodium tripolyphosphate; wherein all proportions are by weight based on the total weight of the composition. The above preferred compositon will normally contain conventional proportions of soil suspending agent, such

as carboxymethyl cellulose, optical brightening agents and perfumes. It preferably contains minor amounts e.g. 0.1 to 2% each of sodium silicate, an alkanolamine such as triethanolamine and/or of phosphonate. It may optionally contain antifoams such as silcones and/or phosphate esters, fabric softeners, such as bentonite, enzymes such as proteases and/or lipases, preferably dispersed in a protective medium such as silicone and preservatives such as formaldehyde.

A further type of preferred composition, where low foam is not a requirement, but where washing efficiency at low temperatures is an important consideration, comprises: water; from 6 to 12 e.g. 7 to 10% sodium linear C_{10-14} alkyl benzene sulphonate; optionally but preferably from 6 to 12 e.g. 7 to 10% sodium C_{14-20} (preferably C_{16-18}) alkyl polyethyleneoxy sulphate having an average of from 1 to 5 e.g. 2 to 4 ethyleneoxy groups per molecule; from 1 to 8% preferably 2 to 7% of C_{8-15} (preferably C_{9-11}) alcohol ethoxylate having an average of from 1 to 5.5. e.g. 2 to 5 ethylene oxide groups per molecule; and from 15 to 30%, preferably 19 to 26% of sodium tripolyphosphate. The composition preferably contains the usual minor ingredients, such as soil suspending agent, optical brightner and perfume, in conventional amounts.

The invention will be illustrated by the following examples, each of which was made by mixing the ingredients in the percentages by weight based on the total composition in the following tables, with the balance in each case water. In each case the surface active ingredients i.e. those components listed in the tables down to and including the C_{16-18} alcohol in the case of examples 1 to 18, were mixed together with sufficient water to form pourable, turbid mixtures having active concentrations between 30 and 50% by weight. In each case samples were centrifuged at 20,000g for 90 minutes to determine the optimum concentrations, at which no separation could be observed.

The resulting pourable non-separating aqueous spherulitic or lamellar compositions were storable. The aqueous active mixtures could also be perfumed and/or combined with the appropriate proportion of carboxymethyl cellulose and/or optical brightening agent and/or phosphonate, with adjustment of concentration when required.

Finally the active concentrate was mixed with the builder (sodium tripolyphosphate, and/or zeolite) and sufficient water to provide the desired final composition.

Each of examples 1 to 18 provided a stable non-sedimenting spherulitic composition having foaming characteristics suitable for use in front loading washing machines and giving good washing performance with both natural and synthetic fibres.

The compositions gave moderate, controlled foaming during the wash cycle and little or no foaming during the rinse cycles.

Eamples 19 to 25 all gave non-sedimenting spherulitic compositions with good low temperature wash performance.

Table 1

Example No	1	2	3	4	5	6	7	8	9	10
Sodium C_{10-14} linear alkyl benzene sulphonate	5.0	4.8	5.5	5.4	5.5	5.8	5.5	5.5	5.0	5.0
C_{12-15} alcohol 3 mole ethoxylate	2.0	1.9	3.2	2.9	3.0	3.3	4.0	4.0	1.0	2.0
Mixed mono and di C_{16-18} alkyl phosphate	0.8	1.9	2.2	2.0	2.0	2.2	2.0	2.0	1.0	1.0
C_{16-18} alcohol	0.8								1.0	1.0
Sodium Tripolyphosphate	30.0	23.8	28.1	24.4	25.0	27.2	20.0	20.0	31.0	30.0
Zeolite A.		4.8		2.4			7.3	7.3		
Optical Brightening agent.	0.15	0.19	0.23	0.20	0.20	0.20	0.20	0.20	0.15	0.15
Sodium Carboxymethyl Cellulose.	0.1	0.1	0.11	0.1	0.1	0.1	0.1	0.1		
Sodium ethylenediamine tetrakis (methenophosphonate)	0.5	0.48	0.56	0.5	0.5	0.5	0.5	1.0	0.5	0.5
Perfume.	0.35	0.38	0.45	0.4	0.4	0.4	0.4	0.4	0.35	0.35

Table 2

Example No.	11	12	13	14	15	16	17	18
Sodium C_{10-14} Linear alkyl benzene Sulphonate.	6.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
C_{12-15} alcohol 3 mole ethoxylate.	2.0						1.0	1.5
Nonyl phenol, 9 mole ethoxylate.			1.0		1.5	1.0	1.5	
Nonyl Phenol, 12 mole ethoxylate.					1.0			
C_{12-18} alcohol, 8 mole ethoxylate.		2.0	1.0					
Mixed mono and di C_{16-18} alkyl phosphate.	1.0	2.0	2.0	2.0	2.0	2.0	1.7	1.7
C_{16-18} alcohol.	1.0							
Sodium Tripolyphosphate.	27.0	28.0	30.0	28.0	28.0	28.0	25.0	25.0
Zeolite A.							4.7	4.7
Optical Brightening agent	0.15	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Sodium Carboxymethyl Cellulose	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sodium ethylenediamine tetrakis (methenophosphonate).	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Perfume.	0.35	0.4	0.4	0.4	0.4	0.4	0.4	0.4

Table 3

Example No.	19	20	21	22	23	24	25
Sodium C_{10-14} linear alkyl benzene sulphonate	6.0	8.96	8.98	7.96	6.99	9.0	7.0
Triethanolamine lauryl sulphate	1.5						
Sodium lauryl 3 mole ethoxysulphate		8.95	8.96	7.98	6.98		
C_{12-13} alcohol 3 mole ethoxylate	2.0						
C_{9-11} alcohol 2.5 mole ethoxylate		2.05		2.08	2.04	3	3
C_{9-11} alcohol 5 mole ethoxylate			2.23	2.22	2.20		
C_{9-11} alcohol 6 mole ethoxylate					2.08		
Sodium tripolyphosphate	25.9	19.86	19.9	19.9	19.9	25	25
Sodium silicate (Na O:SiO_2 = 2.5:1)	0.25						
Sodium ethylenediamine tetrakis (methylenephosphate)	1.5						
Sodium carboxymethyl celluolose	0.1	0.1	0.1	0.1	0.1	0.1	0.1
"TINOPAL" LMS-X optical brightener	0.15						
"TINOPAL" CBS-X optical brightener	0.05	0.1	0.1	0.1	0.1	0.1	0.1
Formaldehyde	0.05						
"ALKALASE" 3.5SL Type WB protease dispersed in silicone	0.2						
Potassium carbonate		1.98	2	2	1.99		
Perfume	0.15-0.3	0.35	0.29	0.29	0.27	0.3	0.3
S132 antifoam	0.2						

Claims

1. A surfactant system for use in a liquid detergent consisting essentially of: an active mixture of (a) at least 50% by weight, based on the total weight of the surfactants present, of anionic surfactants comprising at least 20% by weight, based on the total weight of the surfactants present, of an alkyl benzene sulphonate and (b) at least 2% by weight, based on the total weight of surfactants present of an alkoxylated non-ionic surfactant having an HLB below 12; and sufficient water to provide a non-separating, pourable, spherulitic or G phase composition.

2. A surfactant system according to claim 1 containing less than 10% by weight of the surfactant system, of non-surfactant electrolyte.

3. A surfactant system according to claim 2 wherein the proportion of non-surfactant electrolyte is less than 5% by weight, based on the total weight of the surfactant system.

4. A surfactant system according to any foregoing claim substantially free from organic solvents.

5. A surfactant system according to any foregiong claim containing between 30 and 80% by weight of said active mixture, based on the total weight of the surfactant system.

6. A liquid detergent composition comprising: water; sufficient of an active mixture as specified in claim 1 to provide a total surfactant concentration of from 5 to 25% by weight of the composition; and sufficient, dissolved, surfactant-desolublising electrolyte to provide a non-sedimenting spherulitic composition.

7. A liquid detergent composition according to claim 6 containing suspended solid particles of builder.

8. A composition according to any foregoing claim wherein the alkyl benzene sulphonate is a linear ^{C}12-14 alkyl benzene sulphonate.

9. A composition according to any foregoing claim wherein the anionic surfactant comprises up to 50% by weight based on the total weight of anionic surfactants present, of a ^{C}14-22 mono-and/or di-alkyl phosphate ester.

10. A composition according to claim 9 wherein said alkyl phosphate ester is present in a proportion of 2 to 40% by weight, based on the total weight of anionic surfactant present.

11. A composition according to any foregoing claim wherein the anionic surfactant comprises from 5 to 100% by weight of ^{C}8-20 alkyl sulphate, based on the weight of alkyl benzene sulphonate.

12. A composition according to claim 11 wherein the proportion by weight of alkyl sulphate is from 15 to 30% based on the weight alkyl benzene sulphonate.

13. A composition according to any foregoing claim wherein the anionic surfactant comprises from 5 to 150% by weight of alkyl polyalkyleneoxy sulphate, based on the weight of alkyl benzene sulphonate.

14. A composition according to claim 13 wherein the alkyl polyakylenoxy sulphate is a ^{C}16-18 alkyl 1.5 to 5 mole polyethylenoxy sulphate.

15. A composition according to either of claims 13 and 14 wherein the proportion of alkyl polyalkyleneoxy sulphate is from 50 to 110% by weight, based on the weight of alkyl benzene sulphonate.

16. A composition according to any foregoing claim wherein the nonionic surfactant comprises an ethoxylated alcohol.

17. A liquid detergent composition according to any of claims 6 to 16 comprising suspended solid particles of sodium tripolyphosphate and/or zeolite.

18. A liquid detergent composition according to any of claims 6 to 17 wherein the total proportion by weight of surfactant is from 6 to 12% by weight based on the total weight of the composition.

19. A composition according to claim 18 comprising : water; from 4 to 10% of sodium linear ^{C}10-14 alkyl benzene sulphonate; from 0.5 to 4% of a C10-14 alkyl sulphate salt; from 0.5 to 4% of an ^{C}10-14 alkyl 1 to 5 mole ethoxylate; and from 20-25% sodium tripolyphosphate; all percentages being by weight based on the total weight of the composition.

20. A composition according to claim 19 wherein the alkyl sulphate salt is a triethanolamine salt.

21. A composition according to any of claims 6 to 20 aditionally containing up to 2% each of sodium silicate and a sodium phosphonate and up to 1% each of soil suspending agent and optical brightener.

22. A liquid detergent composition according to any of claims 6 to 17 having from 18 to 25% by weight of total surfactant, based on the total weight of the composition.