



(11) **EP 1 141 221 B2**

(12) **NEW EUROPEAN PATENT SPECIFICATION**
After opposition procedure

(45) Date of publication and mention
of the opposition decision:
30.11.2011 Bulletin 2011/48

(51) Int Cl.:
C11D 17/00 (2006.01) **C11D 3/37** (2006.01)
C11D 3/22 (2006.01)

(45) Mention of the grant of the patent:
17.09.2003 Bulletin 2003/38

(86) International application number:
PCT/EP1999/009377

(21) Application number: **99961040.5**

(87) International publication number:
WO 2000/036079 (22.06.2000 Gazette 2000/25)

(22) Date of filing: **30.11.1999**

(54) **STRUCTURED LIQUID DETERGENT COMPOSITION**

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COMPOSITION DETERGENTE LIQUIDE STRUCTUREE

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

(30) Priority: **16.12.1998 EP 98310336**

(43) Date of publication of application:
10.10.2001 Bulletin 2001/41

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AT BE CH DE DK ES FI FR GR IT LI LU MC NL PT SE
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US-A- 5 168 325 US-A- 5 672 580

Remarks:

The file contains technical information submitted after
the application was filed and not included in this
specification

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Description**FIELD OF THE INVENTION**

5 **[0001]** The present invention is concerned with aqueous liquid detergent compositions of the kind which contain sufficient detergent-active material and, sufficiently dissolved electrolyte to result in a lamellar structure.

BACKGROUND OF THE INVENTION

10 **[0002]** Conventionally, aqueous liquid detergent compositions may be structured in one of two different ways to endow consumer-preferred flow behaviour and/or turbid appearance and/or of suspending particulate solids such as detergency builders or abrasive particles.

15 **[0003]** The first way is to employ an "external structurant" such as a gum or polymer thickener. The second way is to form a lamellar phase "internal structure" from the surfactant(s) and water, the latter usually containing dissolved electrolyte.

20 **[0004]** Lamellar phases are a particular class of surfactant structures which, *inter alia*, are already known from a variety of references, e.g. H.A. Barnes, 'Detergents', Ch.2 in K.Walters (Ed), 'Rheometry: Industrial Applications', J. Wiley & Sons, Letchworth 1980.

25 **[0005]** Lamellar phases can themselves be considered as divided into the sub-classes planar lamellar phases and lamellar droplets. Products can contain exclusively planar lamellar phases or exclusively lamellar droplets or the two forms can co-exist in the same product.

30 **[0006]** The presence of lamellar phases in a liquid detergent product may be detected by means known to those skilled in the art, for example optical techniques, various rheometrical measurements, X-ray or neutron diffraction, and electron microscopy.

35 **[0007]** Lamellar droplets consist of an onion-like configuration of concentric bi-layers of surfactant molecules, between which is trapped water or electrolyte solution (aqueous phase). Systems in which such droplets are close-packed provide a very desirable combination of physical stability and solid-suspending properties with useful flow properties.

40 **[0008]** Examples of internally structured liquids containing a dispersion of lamellar droplets but without suspended solids are given in US patent 4 244 840, whilst examples where solid particles are suspended are disclosed in specifications EP-A-160 342: EP-A-38 101: EP-A-104 452 and also in the aforementioned US 4 244 840. Others are disclosed in European Patent Specification EP-A-151 884, where the lamellar droplets are called 'spherulites'.

45 **[0009]** WO-91/09107 discloses aqueous liquid detergent compositions comprising a dispersion of lamellar droplets of detergent active materials in an aqueous continuous phase.

50 **[0010]** There are also known examples of products containing planar lamellar phases which may be extensive throughout the liquid or distributed as discrete layers interspersed with an aqueous continuous phase. Planar lamellar phases are generally less well suited to combine suspending solid material with preferred flow properties than are lamellar droplets, but they are nevertheless eminently suitable for thickening the product or endowing it with other consumer-preferred properties.

55 **[0011]** Lamellar phases cause the resultant liquid product to be turbid (i.e. cloudy). In order to produce certain visually pleasing effects in aqueous liquid products there is a need to produce a lamellar-structured detergent liquid which is substantially clear (i.e. substantially transparent). Products with a microstructure consisting of predominantly planar lamellar phases are usually less turbid than products with a microstructure of lamellar droplets. However, these products have usually an inhomogeneous appearance and are not substantially clear, so that visually, they do not have a pleasing appearance. Furthermore, in these planar lamellar products, it is often difficult to incorporate sufficient functional electrolytes, e.g. builder or buffer electrolyte, while maintaining clarity. Until now, it has only been possible to produce commercially viable liquid detergents which are substantially clear by use of external structurants in intrinsically isotropic liquids, such as disclosed in GB-A-1 303 810.

SUMMARY OF THE INVENTION

60 **[0012]** A first aspect of the present invention provides an aqueous liquid detergent composition, according to claim 1. Thus, the present invention provides an aqueous detergent composition, comprising a dispersion of lamellar droplets in an aqueous continuous phase, wherein the $D_{v,90}$ of the lamellar droplets is less than 2 micrometers. $D_{v,90}$ = 90% of the volume of all droplets having a diameter smaller than stated.

DETAILED DESCRIPTION OF THE INVENTION

Product Form

[0013] Compositions according to the present invention have a physical form which may be that of a liquid, a pourable gel or a non-pourable gel. These forms are conveniently characterised by the product viscosity. In these definitions, and unless indicated explicitly to the contrary, throughout this specification, all stated viscosities are those measured at a shear rate of 21 s^{-1} and at a temperature of 25°C .

[0014] Compositions according to the present invention which are liquids, preferably have a viscosity of no more than 1,500 mPa.s, more preferably no more than 1,000 mPa.s, still more preferably, no more than 500 mPa.s.

[0015] Compositions according to the present invention which are pourable gels, preferably have a viscosity of at least 1,500 mPa.s but no more than 6,000 mPa.s, more preferably no more than 4,000 mPa.s, still more preferably no more than 3,000 mPa.s and especially no more than 2,000 mPa.s.

[0016] Compositions according to the present invention which are non-pourable gels, preferably have a viscosity of at least 6,000 mPa.s but no more than 12,000 mPa.s, more preferably no more than 10,000 mPa.s, still more preferably no more than 8,000 mPa.s and especially no more than 7,000 mPa.s.

Clarity

[0017] The first aspect of the present invention requires the composition to be substantially clear. This means that the composition has an optical transmissivity of at least 5%, preferably 10%, more preferably 25%, especially $>50\%$, through a path length of 1cm at 25°C .

[0018] These measurements may be obtained using a Perkin Elmer UV/VIS Spectrometer Lambda 12 or a Brinkman PC801 Colorimeter at a wavelength of 520nm, using water as the 100% standard.

[0019] The clarity of the compositions according to the first aspect of the present invention does not preclude the composition being coloured, e.g. by addition of a dye, provided that it does not detract substantially from clarity. Moreover, an opacifier could be included to reduce clarity if required to appeal to the consumer. In that case the definition of clarity applied to the composition according to the invention will apply to the base (equivalent) composition without the opacifier.

Other Visible Solids

[0020] As already mentioned, structuring can be used to suspend particulate solids such as detergency builder or abrasive particles. Normally, these are so small as to simply give the composition a cloudy appearance. However, in compositions according to the present invention, a relatively small number of large particles of functional materials could be suspended to give a pleasing visual effect without affecting the clarity of the bulk of the liquid.

[0021] However, it is also possible to suspend within compositions of the present invention, particles or speckles, purely for their visual effect. These particles, may be coloured. Such particles or speckles may for example be chosen from any of those previously known in liquid detergent products, albeit not in substantially clear internally structured liquids.

[0022] An example of such speckles in externally structured liquids is described in GB-A-1 303 810 which discloses a pourable cleaning or rinsing aqueous detergent compositions in which a visually distinct component is incorporated in the form of particles of at least 500nm in diameter. These particles comprise an agent having a useful effect in the wash, encapsulated in an inert carrier such as wax or gelatin. In order to keep the particles in suspension, the composition comprises a suspending aid such as a gum or a clay.

[0023] An example of a (non-clear) internally structured liquid which contains visible particles or speckles is disclosed in GB-A-2 194 793. The visible particles contain a carrier material such as sodium tripolyphosphate and/or a bentonite clay, plus a pigment. Preferably, these speckles have an average particle size of from 1 to 1000nm (most preferably no more than 100nm), and constitute from 0.5% to 15% by weight of the composition, most preferably from 1% to 5%.

[0024] GB-A-2 247 028 describes a lamellar structured aqueous detergent liquid which is also not substantially clear but in which are dispersed particles or droplets of a sparingly water-soluble or substantially water-insoluble dye.

[0025] It is also possible to utilise coloured speckles or particles of a kind previously proposed for dispersion in a non-aqueous detergent liquid. These are described in EP-0 635 569, according to which the speckles as particles comprising a carrier material such as a bleach, builder, clay, abrasive, enzyme or biopolymer with a dye or pigment associated thereto. These particles must have a $D(3,2)$ average particle size of from 50nm to less than 500nm

[0026] A more recent (unpublished) proposal in the field of internally structured liquids is for an aqueous liquid detergent composition which are not substantially clear but which comprise a structured lamellar phase comprising surfactant, the lamellar phase being capable of suspending particulate solids and being dispersed in a continuous phase, and coloured particles suspended by said lamellar phase, wherein the coloured particles comprise a polymer shell in which is contained a core material, the coloured particles further comprising a colourant.

[0027] According to this unpublished proposal, the colourant of the coloured particles may be contained in the shell and/or the core. The colourant may comprise a dye and/or a pigment material and (as appropriate) may be admixed with, dispersed in and/or

dissolved in the core material and/or the polymer shell material. When colourant is included in the core (whether or not also in the shell), the amount of colourant is preferably from 0.01% to 2%, more preferably from 0.1% to 1% by weight of the total of colourant plus core material. When colourant is additionally or alternatively part of the shell, then it preferably is included at from 0.01% to 4% by weight of the total of colourant plus shell, more preferably from 0.1% to 1% by weight.

[0028] The core material preferably constitutes from 10% to 99%, more preferably from 30% to 98% by weight of the coloured particles.

[0029] The polymer shell may comprise any polymer which is substantially insoluble in the rest of the composition, preferred examples of suitable polymers include poly oxymethylene melamine urea (PMMU), polyamides, cellulosic polymers, poly vinyl alcohol (PVA), Polyurethane and carrageen (i.e. 3, 6-anhydro-d-galactan, which is a polysaccharide), amongst others.

[0030] Suitable core materials include diethylphthalate, alginate, and paraffin oil.

[0031] The D(3,2) average diameter of the coloured particles is from 250 to 2,500 μm , more preferably from 300 to 2,200 μm and most preferably from 350 to 2,000 μm .

[0032] For optimum dispersion within the body of the liquid detergent composition, it is preferred that the average density of the coloured particles is between $\pm 35\%$, more preferably $\pm 30\%$ or $\pm 25\%$, still more preferably $\pm 20\%$, yet more preferably $\pm 18\%$ and most $\pm 15\%$ of the density of the composition without the coloured particles.

[0033] Another means of enhancing the visual appearance of compositions according to the present invention is to incorporate encapsulates of functional materials, e.g. enzymes, which encapsulates may or may not be coloured. However, they will normally be large enough to be distinctly visible so that the bulk of the liquid between the particles still appears substantially clear.

Enzyme encapsulates

[0034] Suitable enzyme encapsulates of the kind mentioned above are intended to effectively protect the enzyme from the adverse effect of UV radiation. Therefore it is essential that the enzyme is adequately contained in the encapsulate to prevent any significant leaking of the enzyme into the liquid detergent composition during storage i.e. preferably less than 50%, more preferably less than 40%, most preferably less than 30% of the encapsulated enzyme leaks into the liquid detergent composition while being stored for 4 weeks at 37°C.

[0035] The enzyme encapsulate may contain polymeric material, but this is not a limiting condition. If polymers are present in the capsules, at least part of the polymeric material should not dissolve in the liquid detergent, whereas they disperse or dissolve upon dilution. Examples of synthetic polymeric materials are:

- polyvinyl alcohol (PVA) of different molecular weight and degrees of hydrolysis, which is defined as a homopolymer or copolymer in which vinyl acetate is a starting monomer unit and in which most or all of the acetate moieties are subsequently hydrolysed to alcohol moieties. (e.g. Airvol range from Air Products, Mowiol range from Hoechst)
- polyamide (obtained via reaction between a diamine with a dicarboxylic acid)
- polyester (obtained via reaction between a diol and a dicarboxylic acid)
- polyurea
- polyurethane
- epoxy resin

[0036] Other examples of natural polymers include:

- methyl cellulose (e.g. Methocal A15LV® ex Dow Chemical)
- hydroxypropylcellulose (e.g. Klucel L® or Klucel G® ex Aqualon)
- hydroxypropylmethylcellulose
- carrageenan (kappa or iota forms) (various types ex FMC)
- alginate (e.g. Manucol DM® or DH ex Kelco).
- gellan gum (e.g. Kelcogel® ex Kelco)
- gelatine

[0037] Further reference: Encyclopaedia of polymers and thickeners for cosmetics, vol. 108, May 1993, 95-135.

[0038] If polymers are present in the capsules, they can be present as a small solid grains, either dispersed throughout the particle or preferentially located in part of the capsules, for example in the outside layer of the capsule. The polymers can also be present as hydrated particles, which can either be dispersed throughout the particle or located in a part of

the capsule. Polymers can also be present in the core of the capsules, in the form of an onion ring inside the capsule or as a shell around the core.

[0039] The enzyme capsules can also contain hydrophobic or fatty materials. Examples of this are:

- 5 - Paraffins (preferably petroleum jelly)
- Triglycerides
- Fatty acids
- Fatty alcohols
- Mixtures of fatty acid and fatty acid soaps
- 10 - Esters (e.g. ceto stearyl stearate)

[0040] If the capsules contain hydrophobic materials they should protect the enzyme against moisture. The hydrophobic material should envelope the enzyme solid particles or droplets which are present in the capsule.

[0041] The capsules can contain also other ingredients:

- 15 - density modifiers, e.g. sucrose
- structurants, e.g. silica, or zeolite
- fillers, e.g. talc, bentonite
- scavengers, e.g., ammonium sulphate
- 20 - plasticizers
- anti-agglomeration or layering agents
- releasing agents

[0042] Thus, in one preferred embodiment the enzyme encapsulate may comprise polymeric material. Preferably, the enzyme encapsulate comprise polymeric materials selected from the group consisting of polyvinylalcohol, polyamide, polyester, polyurea, polyurethane, epoxyresin, methylcellulose, hydroxypropylcellulose, hydroxypropylmethylcellulose, carrageenan, alginate, gellan gum, gelatine and mixtures thereof.

[0043] Examples of enzyme encapsulates can be found in WO-93/07263, EP-A-585295, EP-A-356239, US-A-5 281 356, US-A-5 281 355 and GB-A-2 186 884.

[0044] The enzyme encapsulates have a D (3,2) average diameter between 30 and 5000 μm , preferably between 200 and 3000 μm , most preferably between 500 and 2500 μm . The particle shape can vary from irregular to spherical; in the preferred form they should be close to spherical, but this should not be limiting.

[0045] The enzyme encapsulates have an enzyme encapsulate's density - as measured in the detergent solution - of between 700 and 2500 kg/m^3 , more preferably between 800 and 2000 kg/m^3 and most preferably between 900 and 1500 kg/m^3 .

[0046] The enzyme can be distributed homogeneously throughout the particle (matrix capsule), be located in the core of the capsule (core-shell capsule) or be present in any other confined zone in the capsule, e.g. in an onion-ring shaped zone.

[0047] The enzyme can be present in the capsules in solid form, as small particles, which can comprise pure protein or optionally a mixture of protein and other materials (optionally in a matrix with other components). The enzyme can also be present in the capsule in the form of small droplets of an enzyme solution, or as mixture of solid and liquid (slurry).

[0048] The enzyme encapsulate may comprise any detergent enzymes including protease lipase, amylase, peroxidase, cellulase or a mixture thereof.

[0049] Examples of protease are commercially available types such as Alcalase[™], Durazym[™], Relase[™], Savinase[™] ex Novo Nordisk and Optimase[™], Purafect[™], Properase[™] ex Genencor International.

[0050] Examples of lipase are Lipolase[™] ex Novo Nordisk and Lipomax[™] ex Genencor International.

[0051] Examples of cellulase are Celluzyme[™] and Carezyme[™] ex Novo Nordisk, and Clazinas[™] ex Genencor International. Examples of amylase are Termamyl[™] ex Novo Nordisk and Maxamyl[™] ex Genencor International.

[0052] Preferably the enzyme is a protease.

[0053] When the enzyme is a protease, the protein content is preferably in the range between 0.1 and 20%, more preferably between 0.5% and 10%, most preferably between 1% and 5%.

[0054] When the enzyme is a protease, the enzyme activity is from 100 GU/mg and 20000 GU/mg, more preferably between 500 and 10000 GU/mg, most preferably between 1000 and 5000 GU/mg.

55 Deflocculating Polymer

[0055] In accordance with the second aspect of the present invention, the clarity may be increased by (when the lamellar phase comprises lamellar droplets) incorporating a deflocculating polymer.

[0056] According to the specification of EP-A-346 995, the dependency of stability and/or viscosity upon volume fraction is favourably influenced by incorporating into the lamellar dispersion, a deflocculating polymer comprising a hydrophilic backbone and one or more hydrophobic side-chains.

[0057] The theory of function of these deflocculating polymers is that the hydrophobic chains are anchored in the outer bilayer of the lamellar droplet. The hydrophilic part is extended outwards. These hydrophilic 'brushes' are responsible for the steric stabilisation of the droplets, provided that the 'brushes' exceed a certain length. For surfactant blends in common use, the optimum length of the polymer hydrophobic chain, in order to be anchored into the bilayer is in the order of C_{12} - C_{15} , about the length of the surfactants in the droplet.

[0058] Thus, it is already well known to incorporate deflocculating polymers in aqueous liquid detergents which are structured with lamellar droplet dispersions. However, in these conventional compositions, the polymer is incorporated in a base composition (i.e. the same composition without the polymer) which is already stable and pourable. EP-A- 346 995 defines, in practical terms, the conventional deflocculating effect as that of a polymer in a stable and pourable composition whereby the equivalent composition minus the deflocculating polymer, has a significantly higher viscosity and/or becomes unstable.

[0059] In contrast, compositions according to the present invention are preferably such that the equivalent composition at 25°C, without deflocculating polymer does not have a significantly higher viscosity and is stable.

[0060] Preferably, the term "does not have significantly higher viscosity" means that at a shear rate of $21s^{-1}$, the difference in viscosity is no more than 500 mPa.s, preferably no more than 250 mPa.s.

[0061] Preferably, the term "stable" means that the composition yields no more than 2% by volume visible phase separation when stored at 25°C for 21 days from the time of preparation, more preferably less than 0.1% by volume visible phase separation when stored at 25°C for 90 days from the time of preparation. Compositions according to the present invention in any aspect are preferably "stable" according to these definitions.

[0062] Thus, when any composition according to the present invention comprises deflocculating polymer this may comprise one or more deflocculating polymer materials according to EP-A 346 995 and/or as recited hereinbelow.

[0063] Generally, the amount of material of deflocculating polymer in a composition according to the invention will be from 0.01% to 5.0% by weight of the composition, most preferably from 0.1% to 2.0%.

[0064] For example, EP-A-438 215 discloses preparation of acrylic acid telomers with a functional terminal group, using a secondary alcohol chain transfer agent which may, for example be a C_6 - C_{12} monofunctional secondary alcohol. These materials are described as detergent additives, in particular sequestrants or anti-precipitants. The materials are produced using polymerisation initiators such as ditertiary butyl peroxide. In the description of various different possible initiators, there is mentioned lauryl peroxide.

[0065] Some specific kinds of deflocculating polymers which contain only one hydrophobic moiety and which is attached to an end position of a hydrophilic chain, are disclosed in EP-A-623 670.

[0066] Various sub-types are described for the deflocculating polymers in EP-A-623 670. However, many of those actually exemplified are thiol polyacrylates, that is to say, materials formed by polymerisation of acrylic acid in the presence of a hydrophobic chain transfer agent having from five to twenty five carbon atoms and a terminal-SH group, in a radical polymerisation process. Analogous materials having a thia linkage between the hydrophilic and hydrophobic parts of the molecule are disclosed in US-A-5 489 395, US-A-5 489 397 and EP-A-691 399.

[0067] Another class of suitable deflocculating polymers comprises oligomers or polymers of formula (I) as disclosed in our unpublished international patent application WO 98/55576



wherein Q^1 - represents a hydrophobic moiety, $-X^1$ - and $-Y^1$ -are independently each absent or represent a suitable linking group, $-Z$ - represents a hydrophilic chain; and $-W$ represents hydrogen or a group of formula $-Y^2-X^2-Q^2$, each of $-X^2$, $-Y^2$ and $-Q^2$ being independently selected from the values for X^1 , Y^1 and Q^1 as hereinbefore defined.

[0068] Preferably Q^1 represents an optionally substituted C_5 - C_{30} alkyl, C_5 - C_{30} alkenyl or C_5 - C_{30} aralkyl group, or a hydrophobic monomer residue, such as from lauryl methacrylate or a hydrophobically modified TEMPO (2,2,6,6-tetramethylpiperdiny-1-oxy) moiety. Alkyl, alkenyl or aralkyl groups most preferably have from 8 to 18 carbon atoms and are preferably straight-chained or have only limited branching. Preferably, X^1 is absent or represents a group of formula $(-CH_2-)_n$ where n is 1 or 2 or X^1 is phenyl. Preferably,

Y^1 is absent or represents a carbonyl group, an ester linkage, a hydroxy C_{1-5} alkyl group or a silyl group of formula $(-SiR^1R^2)$, where R^1 and R^2 independently represent $-CH_3$ or $-C_2H_5$; or else Y^1 is a thia-, aza-, carboxy- (i.e. ester), carboxy-aza-, phosphoryl-, phosphonyl- or phosphinyl- linkage, but then with the proviso that W is not hydrogen.

[0069] The group $-Z$ - is preferably a linear, branched or slightly crosslinked molecular composition containing one or more types of relatively hydrophilic monomer units. Preferably the hydrophilic monomers themselves are sufficiently

water soluble to form at least a 1% by weight solution when dissolved in water. The only limitations to the structure of -Z- are that the resultant polymer of formula (I) must be suitable for incorporation in an active-structured aqueous liquid detergent composition and that a polymer corresponding to the hydrophilic moiety alone, i.e. H-Z-H is relatively soluble in water, in that the solubility in water at ambient temperature and at a pH of 3.0 to 12.5 is preferably more than 1 g/l, more preferred more than 5 g/l, most preferred more than 10 g/l.

[0070] Preferably the group -Z- is predominantly linear; more preferably the main chain of the backbone constitutes at least 50% by weight, preferably more than 75%, most preferred more than 90% by weight of the backbone.

[0071] The group -Z- is generally composed of monomer units, which can be selected from a variety of units available for the preparation of polymers.

[0072] Examples of types of monomer units for inclusion alone or in combination in -Z- are:

(i) Unsaturated C₁-C₆ acids, ethers, alcohols, aldehydes, ketones or esters. Preferably these monomer units are mono-unsaturated. Examples of suitable monomers are acrylic acid, methacrylic acid, maleic acid, crotonic acid, itaconic acid, aconitic acid, citraconic acid, vinyl-methyl ether, vinyl sulphonate, vinylalcohol obtained by the hydrolysis of vinyl acetate, acrolein, alkenyl alcohol and vinyl acetic acid. The corresponding salts, e.g. alkali metal salts such as the sodium salt, are also included.

(ii) Cyclic units, either being unsaturated or comprising other groups capable of forming inter-monomer linkages. In linking these monomers the ring-structure of the monomers may either be kept intact, or the ring structure may be disrupted to form the backbone structure. Examples of cyclic monomer units are sugar units, for instance saccharides and glucosides; alkoxy units such as ethylene oxide and hydroxy propylene oxide; and maleic anhydride.

(iii) Other units, for example glycerol, polyalkylene oxide(s) or unsaturated polyalcohol(s) .

[0073] Each of the above mentioned monomer units for inclusion in -Z- may be substituted with groups such as amino, ammonium, amide, sulphonate, sulphate, phosphonate, phosphate, hydroxy, carboxyl and oxide groups.

[0074] The group -Z- is preferably composed of one or two monomer types but also possible is the use of three or more different monomer types in one hydrophilic backbone. Examples of preferred hydrophilic backbones are:

homopolymers of acrylic acid, copolymers of acrylic acid and maleic acid, poly (2-hydroxy ethyl acrylate), polysaccharides, cellulose ethers, polyglycerols, polyacrylamides, polyvinylalcohol/polyvinylether copolymers, poly sodium vinyl sulphonate, poly 2-sulphato ethyl methacrylate, polyacrylamido methyl propane sulphonate and copolymers of acrylic acid and trimethylol propane triacrylate.

[0075] Optionally, the group -Z- may also contain small amounts of relatively hydrophobic units, e.g. those derived from polymers having a solubility of less than 1g/l in water, provided that the overall solubility of the hydrophilic polymer backbone still satisfies the solubility requirements as specified hereabove. Examples of relatively water insoluble polymers are polyvinyl acetate, polymethyl methacrylate, polyethyl acrylate, polyethylene, polypropylene, polystyrene, polybutylene oxide, polypropylene oxide and polyhydroxy propyl acetate.

[0076] Preferred sub-classes of the oligomers or polymers of formula (I) (hereinafter referred to as "materials of the invention"), include respectively, those where W is hydrogen, those where W is -Y²-X²-Q², some or all of X², Y² and Q² respectively differing from X¹, Y¹ and Q¹ and those where W is -Y²-X²-Q², X², Y² and Q² each being the same as X¹, Y¹ and Q¹.

[0077] If W is hydrogen, there is only a single hydrophobic moiety attached to one end of the hydrophilic moiety. Such materials are ideally suited as deflocculating materials. If W is a group -Y²-X²-Q² then there is a respective hydrophobic group at either end of the hydrophobic moiety. Such materials may be employed for deliberate bridging of lamellar droplets, e.g. to increase viscosity.

[0078] Of course, as mentioned above, often deflocculation is needed to inhibit viscosity increase at high volume fractions so that in principle, bridging can be undesirable. However, the bridging materials having a pair of hydrophobic groups (W not hydrogen) are within the ambit of the present invention. For example, a predetermined blend of materials of the invention may be used, comprising one deflocculating material to control stability and one bridging material to increase viscosity in a controlled fashion.

[0079] The bridging material has, on average, more than one hydrophobic (Q¹/Q²) groups per molecule and preferably two or more such hydrophobic groups. As a consequence the molecular weight (M_w) of the bridging material is larger than (x.M_i + M_o), preferably larger than (x.M_i + 2M_o) and more preferably larger than 2(x.M_i + M_o), with x being the molecular ratio between hydrophilic monomers and hydrophobic monomers, M_i being the average molecular weight of the hydrophilic groups and M_o the average molecular weight of the hydrophobic groups.

[0080] The bridging polymer is preferably prepared using conventional aqueous polymerisation procedures, but em-

playing a process wherein the polymerisation is carried out in the presence of a suitable cosolvent and wherein the ratio of water to cosolvent is carefully monitored so as to keep the polymer as it forms in a sufficiently mobile condition and to prevent unwanted homopolymerisation and precipitation of the polymer from the hydrophobic monomer. The process of the invention provides a product which is stable and clear and which exhibits no gelling or product separation on standing. Suitable cosolvents are selected from the group consisting of isopropanol, n-propanol, acetone, lower (C₁ to C₄) alcohols, esters and ketones and wherein the water to cosolvent ratio is smaller than 1.5, more preferably less than 1.0, more preferably less than 0.75, and especially less than 0.5.

[0081] The use of a better defined mixture of a deflocculating material and of a bridging material allows a degree of control of rheology not possible with the "cocktail" of polymers resulting from the process of EP-A-346 995.

[0082] Nevertheless, it should be appreciated that any method of forming either the deflocculating (W=H) or bridging (W= -Y²-X²-Q²) oligomers or polymers of formula (I) will not form 100% pure materials. However, sample oligomers or polymers according to the present invention will have a high weight percentage of oligomer or polymer species having a structure of formula (I), although not necessarily all of that percentage will have the same structure of formula (I). Thus, a preferred sample or batch of oligomer and/or polymer material the present invention may have at least 50% by weight of its total of oligomers and/or polymers having the general formula (I) as defined in claim 1, or optionally, of any preferred sub-class of polymers or oligomers of formula (I) as defined in the description or any other claim. This weight percentage is more preferably, in ascending order of preference, at least 65%, 70%, 75%, 80%, 85% or 90% by weight of the total batch or sample.

Lamellar Droplets

[0083] The present invention provides compositions whereby the lamellar phase comprises lamellar droplets.

[0084] The present invention relies on the finding of being able to produce clarity by limiting the size of a significant fraction of the lamellar droplets, i.e. so that their D_{v,90} is less than 2 μm, more preferably less than 1.0 μm, e.g. less than 0.5 μm, still more preferably less than 0.2 μm, yet more preferably less than 0.1 μm and especially less than 0.05 μm. The D_{v,90} of the droplets is defined as 90% of the volume of all droplets having a diameter smaller than that indicated. The actual value of D_{v,90} for a given sample may be determined by making electron microscopy pictures of the liquid detergent composition at a magnification of between 15,000 and 60,000 (preferably about 30,000) and determining the relative number of droplets of each diameter and calculating from the obtained cumulative diameter size distribution the cumulative volume size distribution or by laser light scattering particle sizers such as the Malvern Mastersizer.

[0085] The D_{v,90} of the droplets can be brought to below the critical value, for example by incorporating deflocculating polymer, or by using as part of the surfactant blend, so-called stabilising surfactants as disclosed in EP-A-328 177. Other ways of producing small lamellar droplets of the defined size include processing routes where high shear conditions are used to apply high fluid stresses. This will be explained in more detail hereinbelow in the section relating to processing.

Refractive Index

[0086] In accordance with the present invention it is preferred that the refractive index of the lamellar phase and that of the aqueous phase are substantially matched in such a way that the composition has an optical transmissivity of at least 5%.

[0087] The refractive index of the lamellar phase (n_{lam}) can be calculated by using the refractive index of each component (n_k) in the lamellar phase and the volume fraction (v_k/v_{lam}) by which that component is present in the lamellar phase using:

$$\frac{n_{lam}^2 - 1}{n_{lam}^2 + 2} * V_{lam} = \sum_{k=1}^n \frac{n_k^2 - 1}{n_k^2 + 2} * V_k$$

[0088] The refractive index of the liquid detergent composition as a whole can for example be determined as follows. Light having a wavelength of 589 nm is passed through a thin layer (preferably about 1 mm) of liquid detergent composition. The angle of incidence and the angle of refraction are measured, whereafter the refractive index can be calculated by using the Snellius equation. Another, preferred method to determine the refractive index is by using internal reflection measurements, for example by using an Atago digital refractometer RX-1000 or a Bellingham and Stanley refractometer RFM91. The use of internal reflection measurements is especially advantageous for determining the refractive index for opaque systems.

[0089] The refractive index of the corresponding aqueous phase can be measured by isolating the aqueous phase

from the detergent composition (e.g. by (ultra-) centrifugation) or by separate preparation of a composition, whereby the insoluble ingredients are only added to their solubility limit and the dispersed phases are omitted.

[0090] Preferably, the difference between the refractive index of the lamellar phase and the aqueous phase is no greater than 0.02, more preferably no greater than 0.01, still more preferably no greater than 0.005 and especially no greater than 0.002.

[0091] To achieve substantial refractive index matching, the refractive index of the aqueous phase can be increased and/or the refractive index of the lamellar phase can be decreased.

[0092] The refractive index of the aqueous phase may be increased by dissolving therein materials. However, often the added materials which results in a refractive index increase, affect the physical stability of the system, like electrolyte or hydrotrope. Other relatively low molecular weight materials may not significantly affect the stability or viscosity of the composition, although there will be some effect because being an additional component of the aqueous phase, it will inevitably affect the volume fraction of the lamellar phase and/or the viscosity of the aqueous phase. Often these components are neutral non-electrolytic materials of relatively low molecular weight.

[0093] The water soluble non-electrolyte materials for increasing the refractive index of the aqueous phase is sugar.

[0094] Suitable sugars include mono saccharides such as glucose and fructose, disaccharides such as saccharose, sucrose, lactose, maltose and cellobiose. Glucose syrups can also be employed. These contain mixtures of mono, di and polysaccharides. Preferably the mono and disaccharide fractions of the carbohydrate mix should be at least 50%. As mentioned above, it is already known to use non-sugar polyols such as glycerol or sorbitol, in minor amounts in aqueous liquid detergents, for enzyme stabilisation. Such materials may additionally be employed in the compositions according to the present invention for refractive index matching but the amounts will be higher than for enzyme stabilisation, e.g. as specified hereinbelow.

[0095] Also useful for refractive index matching, although less preferred, are polysaccharides such as water soluble gums, e.g. guar gum, xanthan gum, arabic gum and tragacanth, because these ingredients increase the viscosity of the total system when added in appreciable additional amounts for refractive index matching.

[0096] A further class of additionally useful materials to increase the refractive index of the aqueous phase are polyols, such as glycerol and polyethylene glycol.

[0097] The amount of water soluble non-electrolyte material in the composition will be chosen as that required to effect the substantial refractive index matching. However the minimum will amount typically be 2.5%, preferably 5%, especially 10%, by weight of the total composition. The maximum amount of water soluble non-electrolyte material is typically 50%, preferably 40%, especially 30% by weight of the total composition. If it is desired to specify a particular range of these amounts, any specified minimum value may be paired with any specified maximum.

Detergent Active Material

[0098] The refractive index of the lamellar phase may be reduced by choosing an appropriate surfactant or blend of surfactants. One suitable approach is to substantially exclude aralkyl surfactants such as alkyl benzene sulphonates, i.e. the total of aralkyl surfactants should be less than 30%, preferably less than 10%, more preferably less than 5%, and especially less than 1% by weight of the total surfactants (including any soap). Most preferably, such aralkyl surfactants are completely absent.

[0099] To formulate a surfactant blend suitable for forming a lamellar phase without using aralkyl materials, one may, for example, employ a blend of primary and/or secondary alkane sulphate or sulphonate material together with one or more nonionic surfactants.

[0100] Examples of suitable alkane sulph(on)ates are sodium and potassium alkyl sulphates, especially those obtained by sulphonating higher (C₈-C₁₈), primary or secondary alcohols produced, for example, from tallow or coconut oil.

[0101] Suitable nonionic surfactants include, in particular, the reaction products of compounds having a hydrophobic group and reactive hydrogen atom, for example aliphatic alcohols, acids, amides with alkylene oxides, especially ethylene oxide, either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl (C₆-C₁₈) primary or secondary linear or branched alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long-chain tertiary phosphine oxides and dialkyl sulphoxides.

[0102] Preferably, the weight ratio at the total alkane sulph(on)ate material to the total nonionic material is from 90:10 to 10:90, more preferably from 80:20 to 50:50.

[0103] Another suitable surfactant blend for this purpose comprises one or more soaps with one or more nonionic surfactants.

[0104] Suitable soaps include alkali metal soaps of long chain mono- or dicarboxylic acids for example one having from 12 to 18 carbon atoms. Typical acids of this kind are oleic acid, ricinoleic acid and fatty acids derived from castor oil, rapeseed oil, groundnut oil, coconut oil, palm kernel oil or mixtures thereof. The sodium or potassium soaps of these acids can be used.

[0105] Suitable nonionic surfactants to blend with the soap are mentioned above. Preferably, the weight ratio of the total soap to the total nonionic material is from 60:40 to 90:10, more preferably from 70:30 to 80:20.

[0106] In other preferred compositions, part or all of the detergent active material is a stabilising surfactant, which has an average alkyl chain length greater than 6 C-atoms, and which has a salting out resistance, greater than, or equal to 6.4. These stabilising surfactants are disclosed in EP-A-328 177. Examples of these materials are alkyl polyalkoxylated phosphates, alkyl polyalkoxylated sulphosuccinates; dialkyl diphenyloxide disulphonates; alkyl polysaccharides and mixtures thereof. The advantage of these surfactants is that they are surfactants with a relatively low refractive index and these surfactants tend to decrease the droplet size of the lamellar droplets.

[0107] Both effects have a positive effect on the clarity of the systems.

[0108] However, aside from any desire to formulate the surfactant content to reduce the refractive index of the lamellar phase, in the widest sense, the detergent-active material in the composition, in general, may comprise one or more surfactants, and may be selected from anionic, cationic, nonionic, zwitterionic and amphoteric species, and (provided mutually compatible) mixtures thereof. For example, they may be chosen from any of the classes, sub-classes and specific materials described in 'Surface Active Agents' Vol. 1, by Schwartz & Perry, Interscience 1949 and 'Surface Active Agents' vol. II by Schwartz, Perry & Berch (Interscience 1958), in the current edition of "McCutcheon's Emulsifiers & Detergents" published by the McCutcheon division of Manufacturing Confectioners Company or in 'Tensid-Taschenbuch', H. Stache, 2nd Edn., Carl Hanser Verlag, München & Wien, 1981.

[0109] In many (but not all) cases, the total detergent-active material may be present at from 2% to 60% by weight of the total composition, for example from 5% to 40% and typically from 10% to 30% by weight. However, one preferred class of compositions comprises at least 15%, most preferably at least 25% and especially at least 30% of detergent-active material based on the weight of the total composition. In the case of blends of surfactants, the precise proportions of each component which will result in such stability and viscosity will depend on the type(s) and amount(s) of the electrolytes, as is the case with conventional structured liquids.

[0110] Common anionic surfactants are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals.

[0111] Aside from anionic surfactants already mentioned with regard to refractive index control, where appropriate, one may still employ conventional sodium and potassium alkyl (C_9 - C_{20}) benzene sulphonates, particularly sodium linear secondary alkyl (C_{10} - C_{15}) benzene sulphonates; sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum. Other suitable anionics include sodium coconut oil fatty monoglyceride sulphates and sulphonates; sodium and potassium salts of sulphuric acid esters of higher (C_6 - C_{18}) fatty alcohol-alkylene oxide, particularly ethylene oxide, reaction products; the reaction products of fatty acids such as coconut fatty acids esterified with isethionic acid and neutralised with sodium hydroxide; sodium and potassium salts of fatty acid amides of methyl taurine; alkane monosulphonates such as those derived by reacting alpha-olefins (C_{8-20}) with sodium bisulphite and those derived from reacting paraffins with SO_2 and Cl_2 and then hydrolyzing with a base to produce a random sulphonate; and olefin sulphonates, which term is used to describe the material made by reacting olefins, particularly C_{10} - C_{20} alpha-olefins, with SO_3 and then neutralising and hydrolyzing the reaction product.

Water

[0112] Preferably the amount of water in the composition is from 5 to 95%, more preferred from 25 to 75%, most preferred from 30 to 50%. Especially preferred less than 45% by weight.

Electrolyte

[0113] The aqueous continuous phase contains electrolyte. As used herein, the term electrolyte means any ionic water-soluble material. However, in lamellar dispersions, not all the electrolyte is necessarily dissolved but may be suspended as particles of solid because the total electrolyte concentration of the liquid is higher than the solubility limit of the electrolyte. Mixtures of electrolytes also may be used, with one or more of the electrolytes being in the dissolved aqueous phase and one or more being substantially only in the suspended solid phase. Two or more electrolytes may also be distributed approximately proportionally, between these two phases. In part, this may depend on processing, e.g. the order of addition of components. On the other hand, the terms 'salts' includes all organic and inorganic materials which may be included, other than surfactants and water, whether or not they are ionic, and this term encompasses the sub-set of the electrolytes (water-soluble materials).

[0114] However, there is a limit to the size and amount of non-dissolved (i.e. suspended) electrolytes in these formulation which is consistent with the objective of clarity. The amount of small particles which are not visible as separate entities should be so low that the bulk of the liquid remains substantially clear in accordance with the definition of the

first aspect of the present invention. The amounts of relatively large particles (i.e. visible as separate entities) should be such that they have a pleasing visual effect like the aforementioned "visible solids".

[0115] The only restriction on the total amount of detergent-active material and electrolyte (if any) is that in the compositions of the invention, together they must result in formation of an aqueous lamellar dispersion. Thus, within the ambit of the present invention, a very wide variation in surfactant types and levels is possible. The selection of surfactant types and their proportions, in order to obtain a stable liquid with the required structure will be fully within the capability of those skilled in the art.

[0116] Preferably, the compositions contain from 1% to 60%, especially from 10 to 45% of a salting-out electrolyte. Salting-out electrolyte has the meaning ascribed to in specification EP-A-79 646. Optionally, some salting-in electrolyte (as defined in the latter specification) may also be included, provided if of a kind and in an amount compatible with the other components and the composition is still in accordance with the definition of the invention claimed herein. Some or all of the electrolyte (whether salting-in or salting-out), or any substantially water-insoluble salt which may be present, may have detergency builder properties. In any event, it is preferred that compositions according to the present invention include detergency builder material, some or all of which may be electrolyte. The builder material is any capable of reducing the level of free calcium ions in the wash liquor and will preferably provide the composition with other beneficial properties such as the generation of an alkaline pH, the suspension of soil removed from the fabric and the dispersion of the fabric softening clay material.

Detergency Builder

[0117] As already mentioned, water soluble inorganic detergency builders (if dissolved in the aqueous phase) are electrolytes but any solid material above the solubility limit will normally be suspended by the lamellar phase.

[0118] Examples of phosphorous-containing inorganic detergency builders, when present, include the water-soluble salts, especially alkali metal pyrophosphates, orthophosphates, polyphosphates and phosphonates. Specific examples of inorganic phosphate builders include sodium and potassium triphosphates, phosphates and hexametaphosphates. Phosphonate sequestrant builders may also be used.

[0119] Examples of non-phosphorous-containing inorganic detergency builders, when present, include water-soluble alkali metal carbonates, bicarbonates, silicates and crystalline and amorphous aluminosilicates. Specific examples include sodium carbonate (with or without calcite seeds), potassium carbonate, sodium and potassium bicarbonates, silicates and zeolites, although there are restrictions with respect to the amount and volume fraction of solid particles which can be added while retaining substantial clarity.

[0120] In the context of inorganic builders, we prefer to include electrolytes which promote the solubility of other electrolytes, for example use of potassium salts to promote the solubility of sodium salts. Thereby, the amount of dissolved electrolyte can be increased considerably (crystal dissolution) as described in UK patent specification GB 1 302 543.

[0121] Examples of organic detergency builders, when present, include the alkaline metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates, polyacetyl carboxylates, carboxymethyloxysuccinates, carboxymethyloxymalonates, ethylene diamine-N,N-disuccinic acid salts, polyepoxysuccinates, oxydiacetates, triethylene tetramine hexa-acetic acid salts, N-alkyl imino diacetates or dipropionates, alpha sulpho- fatty acid salts, dipicolinic acid salts, oxidised polysaccharides, polyhydroxysulphonates and mixtures thereof.

[0122] Specific examples include sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilo-triacetic acid, oxydisuccinic acid, melitic acid, benzene polycarboxylic acids and citric acid, tartrate mono succinate and tartrate di succinate.

[0123] In the context of organic builders, it is also desirable to incorporate polymers which are only partly dissolved in the aqueous continuous phase. This allows a viscosity reduction (owing to the polymer which is dissolved whilst incorporating a sufficiently high amount to achieve a secondary benefit, especially building, because the part which is not dissolved does not bring about the instability that would occur if substantially all were dissolved). As for inorganic builders, the same restrictions apply with respect to the amount and volume fraction of non-dissolved polymer phase which can be added while retaining substantial clarity.

Other Polymers

[0124] Examples of partly dissolved polymers include many of the polymer and co-polymer salts already known as detergency builders. For example, may be used (including building and non-building polymers) polyethylene glycols, polyacrylates, polymaleates, polysugars, polysugarsulphonates and copolymers of any of these. Preferably, the partly dissolved polymer comprises a co-polymer which includes an alkali metal salt of a polyacrylic, polymethacrylic or maleic acid or anhydride. Preferably, compositions with these copolymers have a pH of above 8.0 In general, the amount of viscosity-reducing polymer can vary widely according to the formulation of the rest of the composition. However, typical amounts are from 0.5 to 4.5% by weight.

[0125] It is further possible to include in the compositions of the present invention, alternatively, or in addition to the partly dissolved polymer, yet another polymer which is substantially totally soluble in the aqueous phase and has an electrolyte resistance of more than 5 grams sodium nitrilotriacetate in 100 ml of a 5% by weight aqueous solution of the polymer, said second polymer also having a vapour pressure in 20% aqueous solution, equal or less than the vapour pressure of a reference 2% by weight or greater aqueous solution of polyethylene glycol having an average molecular weight of 6,000; said second polymer having a molecular weight of at least 1,000.

[0126] The incorporation of the soluble polymer permits formulation with improved stability at the same viscosity (relative to the composition without the soluble polymer) or lower viscosity with the same stability. The soluble polymer can also reduce viscosity drift, even when it also brings about a viscosity reduction. Here, improved stability and lower viscosity mean over and above any such effects brought about by the deflocculating polymer.

[0127] It is especially preferred to incorporate the soluble polymer with a partly dissolved polymer which has a large insoluble component. That is because although the building capacity of the partly dissolved polymer will be good (since relatively high quantities can be stably incorporated), the viscosity reduction will not be optimum (since little will be dissolved). Thus, the soluble polymer can usefully function to reduce the viscosity further, to an ideal level.

[0128] The soluble polymer can, for example, be incorporated at from 0.05 to 20% by weight, although usually from 0.1 to 10% by weight of the total composition is sufficient, and especially from 0.2 to 3.5 - 4.5% by weight. It has been found that the presence of deflocculating polymer increase the tolerance for higher levels of soluble polymer without stability problems. A large number of different polymers may be used as such a soluble polymer, provided the electrolyte resistance and vapour pressure requirements are met. The former is measured as the amount of sodium nitrilotriacetate (NaNTA) solution necessary to reach the cloud point of 100 ml of a 5% w/w solution of the polymer in water at 25°C, with the system adjusted to neutral pH, i.e. about 7. This is preferably effected using sodium hydroxide. Most preferably, the electrolyte resistance is 10 g NaNTA, especially 15g. The latter indicates a vapour pressure low enough to have sufficient water binding capability, as generally explained in the applicants' specification GB-A-2 053 249. Preferably, the measurement is effected with a reference solution at 10% by weight aqueous concentration, especially 18%.

[0129] Typical classes of polymers which may be used as the soluble polymer, provided they meet the above requirements, include polyethylene glycols, Dextran, Dextran sulphonates, polyacrylates and polyacrylate/maleic acid co-polymers.

[0130] The soluble polymer must have an average molecular weight of at least 1,000 but a minimum average molecular weight of 2,000 is preferred.

[0131] The use of partly soluble and the use of soluble polymers as referred to above in detergent compositions is described in our European patent specifications EP-A-301 882 and EP-A-301 883.

Hydrotropes

[0132] Although it is possible to incorporate minor amounts of hydrotropes such as lower alcohols (e.g. ethanol) or alkanolamines (e.g. triethanolamine), in order to ensure integrity of the lamellar dispersion we prefer that the compositions of the present invention are substantially free from hydrotropes. By hydrotrope is meant any water soluble agent which tends to enhance the solubility of surfactants in aqueous solution.

Other Optional Ingredients

[0133] Apart from the ingredients already mentioned, a number of optional ingredients may also be present. Enzymes, optionally together with enzyme stabilisers may be incorporated. Enzymes in encapsulated form, as a visually distinct suspended component have already been mentioned hereinbefore.

[0134] Other optional ingredients include lather booster such as alkanolamides, particularly the monoethanolamides derived from palm kernel fatty acids and coconut fatty acids, fabric softeners such as clays, amines and amine oxides; lather depressants, oxygen-releasing bleaching agents such as sodium perborate and sodium percarbonate; peracid bleach precursors, chlorine-releasing bleaching agents such as trichloroisocyanuric acid, inorganic salts such as sodium sulphate, and, usually present in very minor amounts, fluorescent agents, perfumes, germicides and colourants, oily-soil release polymers, such as Poly Ethylene Terephthalate - Poly Oxy Ethylene Terephthalates or (partly) sulphonate versions thereof (including Permalose and Aquaperle (Trademarks) ex. ICI, Gerol and Repe-O-Tex (Trademarks) ex. Rhone-Poulenc and Sokalan HP22 (Trademark) ex. BASF); anti-redeposition agents, such as sodium carboxy methyl cellulose; anti-dye transfer agents, such as PVP, PVI and co-polymers thereof.

[0135] Amongst these optional ingredients, as mentioned previously, are agents to which lamellar dispersions without deflocculating polymer are highly stability-sensitive and by virtue of the present invention, can be incorporated in higher, more useful amounts. These agents cause a problem in the absence of deflocculating polymer because they tend to promote flocculation of the lamellar droplets. Examples of such agents are soluble polymers, soluble builders such as succinate builders, fluorescers like Blankophor RKH, Tinopal LMS, and Tinopal DMS-X and Blankophor BBH as well as

metal chelating agents, especially of the phosphonate type, for example the Dequest range sold by Monsanto.

Processing

[0136] Compositions of the invention may be prepared by any conventional method for the preparation of liquids detergent compositions. A preferred method involves the dispersing of the electrolyte ingredient together with the minor ingredients except for the temperature sensitive ingredients (if any) in water of elevated temperature, followed by the addition of the builder material (if any), the detergent active material under stirring and thereafter cooling the mixture and adding any temperature sensitive minor ingredients such as enzymes, perfumes etc. The deflocculating polymer (where used) may for example be added after the electrolyte ingredient or as the final ingredient.

[0137] The manner of preparation and of treatment post processing can materially influence the optical transmittance and clarity of the composition produced. In particular the use of high shear conditions (preferably at least $10,000\text{s}^{-1}$) to apply high fluid stresses and facilitate the production of small lamellar droplets is preferred for the first aspect of the invention. The high shear conditions (where "shear" refers to deformation rates involving either or both shear or extensional deformations) can be applied during the preparation, for example during the formation of lamellar droplets stage.

[0138] Thus, a second aspect of the present invention provides a process for preparing a composition according to the present invention, the process comprising mixing at least some of the ingredients of the composition at a shear rate of at least $10,000\text{s}^{-1}$ and then admixing the resultant composition with any remaining ingredients.

[0139] High fluid stresses and consequent smaller lamellar droplets created by application of high shear during the preparation are enhanced by employing mixes of higher viscosity. To this end, it is preferable to add the sugar component prior to the surfactants in order to thicken the mix at the point of formation of the lamellar droplets. However, due regard has to be given to the chemical sensitivity of the sugar. For example pH sensitive sugars such as fructose should not be exposed to extremes of pH during the preparation.

[0140] Higher viscosity mixes can also be generated by withholding some of the process water in order to apply the high shear step to a concentrate of the final product followed by a dilution step, for example as part of the process described in the specification of patent application WO 96/20270. Alternatively the high shear conditions can be applied to the final composition after preparation. High shear may be applied by a static device, for example a shear valve such as a Saunders diaphragm valve. Preferably it is applied by a dynamic device such as a dynamic mill. Examples of such devices include those manufactured by Silverson, Fryma or Janke & Kunkel and that described in the specification of patent application WO 96/20270. Preferably the shear device should be located in line in order to minimise aeration of the composition which would detract from the optical transmittance and clarity. In the event that the composition becomes substantially aerated a de-aeration step, such as centrifugation, can be incorporated into the preparation.

[0141] The following examples are intended to further illustrate the invention and are not intended to limit the invention in any way:

All percentages, unless indicated otherwise, are intended to be percentages by weight.

All numerical ranges in this specification and claims are intended to be modified by the term about.

[0142] Finally, where the term comprising is used in the specification or claims, it is not intended to exclude any terms, steps or features not specifically recited.

EXAMPLES

[0143] The invention will now be illustrated by way of the following Examples, in all Examples, unless stated to the contrary, all percentages are by weight.

Ingredient	% by weight (if not otherwise stated)			
	Comparative example	Ex. 1	Ex. 2	Ex. 3
Nonionic, ® Synperonic A7®	3.0	3.0	3.0	3.0
Oleic acid, Priolene 6907	7.0	7.0	7.0	7.0
NaOH, to pH ~ 9.0	0.99	0.99	0.99	0.99
STP	12.5	12.5	12.5	12.5
Deflocculating polymer (1)				1.0

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(continued)

Ingredient	% by weight (if not otherwise stated)			
	Comparative example	Ex. 1	Ex. 2	Ex. 3
Water	up to 100%	up to 100%	up to 100%	up to 100%
Saccharose % added on top		20	40	20
Stability	OK	OK	OK	OK
Viscosity (mPa.s at 21 s-1)	150	320	340	690
Optical transmissivity (%) at 520nm	0.2	12	46	66

[0144] These examples show:

- sample without deflocculating polymer is stable
- addition of sugar increases the clarity
- on addition of deflocculating polymer more clarity is obtained

Ingredient	% by weight (if not otherwise stated)					
	Comparative example	Ex. 4	Ex. 5	Comparative example	Ex. 6	Ex. 7
LAS-acid	7.0	7.0	7.0	7.0	7.0	7.0
Nonionic, Synperonic A7®	3.0	3.0	3.0	3.0	3.0	3.0
NaOH, to pH ~ 8.0	0.86	0.86	0.86	0.86	0.86	
STP	11.0	11.0	11.0			0.86
Na-citrate. 2aq				12.5	12.5	12.5
Deflocculating polymer (1)		1.0	1.0		1.0	1.0
Water	up to 100%	up to 100%	up to 100%	up to 100%	up to 100%	up to 100%
Saccharose, % added on top		20	40		10	
Fructose % added on top						40
Stability	Unstable	OK	OK	OK	OK	OK
Viscosity (mPa.s at 21 s-1)	40	550	630	180	600	380
Optical transmissivity (%) at 520nm	0.04	61	67	0.2	9.2	19
Dv 90 (µm)	> 10		0.8			

[0145] These examples show:

- sample without deflocculating polymer is stable
- addition of sugar increases the clarity
- on addition of deflocculating polymer (much) more clarity is obtained
- effect is not restricted to saccharose

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Ingredient	% by weight (if not otherwise stated)					
	Comparative example	Ex. 8	Ex. 9	Ex. 10 Comparative	Ex. 11	Ex.12
LAS-acid	5.0	5.0	5.0	5.0	5.0	5.0
Nonionic, Synperonic A7®	3.0	3.0	3.0	3.0	3.0	3.0
APG, Glucopon 600CS®	2.0	2.0	2.0	2.0	2.0	2.0
NaOH, to pH - 8.5	0.61	0.61	0.61	0.61	0.61	0.61
STP	11.0	11.0	11.0	11.0	11.0	11.0
Deflocculating polymer (1)				1.0	1.0	1.0
Water	up to 100%	up to 100%	up to 100%	up to 100%	up to 100%	up to 100%
Saccharose, % added on top		20	40		20	40
Stability	OK	OK	OK	OK	OK	OK
Viscosity (mPa.s at 21 s-1)	140	210	270	410	480	470
Optical transmissivity (%) at 520nm	0.2	5.2	38	8.8	51	53

Ingredient	% by weight (if not otherwise stated)	
	Comparative example	Ex. 13
LAS-acid	5.0	5.0
Nonionic, ® Synperonic A7	3.0	3.0
APG, Glucopon 600CS®	2.0	2.0
NaOH, to pH ~ 10	0.61	0.61
Na-citrate. 2aq	12.5	12.5
Deflocculating polymer (1)		1.0
Water	up to 100%	up to 100%
Saccharose, % added on top		40
Stability	OK	OK
Viscosity (mPa.s at 21 s-1)	200	590
Optical transmissivity (%) at 520nm	0.13	16

Ingredient	parts by weight (if not otherwise stated)	
	Ex. 14 Comparative	Ex. 15 Comparative
LAS-acid	9.4	14.1

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(continued)

Ingredient	parts by weight (if not otherwise stated)	
	Ex. 14 Comparative	Ex. 15 Comparative
LES	15	10
NaOH	1.15	1.72
Na-citrate. 2aq	17.1	17.1
Deflocculating polymer (2)	0.25	0.25
Water	up to 100%	up to 100%
Stability	OK	OK
Viscosity (mPa.s at 21 s-1)	6170	6980
Optical transmissivity (%) at 520nm	35	10

Raw materials	
LAS-acid	approx C12 alkyl benzene sulphonic acid, ex Huls
LES	Sodium lauryl ether (approx. 3 ethylene oxide) sulfate Manro Bes 70™ ex Hickson Manro
Synperonic A7™	C13-15 alcohol, ethoxylated with 7 EO groups, ex ICI
Oleate	Priolene 6907™, ex Unichema
APG Henkel	Alkyl PolyGlucoside, Glucopon 600CS™ ex
STP	Sodium Tri (Poly) Phosphate, Thermphos NW™, ex Knapsack or Sodium Tri (Poly) Phosphate, Rodiaphos HPA-3.5™, ex Rhone Poulenc
Na-citrate.2aq	Sodium citrate ex Merck
Deflocculating polymer (1)	Polymer All™ from EP 346,995, ex National Starch
Deflocculating polymer (2)	Marchon XB 16™, ex Albright & Wilson
Saccharose	ex Cooperatieve Suiker Maatschappij, The Netherlands
Fructose	ex Merck

[0146] The following examples all illustrate the beneficial influence of applying shear to generate transparency. In examples A and B shear was applied to final product using a Silverson LN4 dynamic mill on maximum setting for 10 minutes. Samples were then centrifuged to remove air.

[0147] Examples 16-19 - various concentrations of glucose + Silverson. Shear rate approximately 50,000 sec⁻¹.

Ingredient	% by weight (if not otherwise stated)									
	Comparative example	Ex. 16	Comparative example	Ex. 17	Comparative example	Ex. 18	Comparative example	Ex. 19	Comparative example	Ex. A5
LAS-acid	7.7	7.7	7.7	7.7	7.7	7.7	7.7	7.7	7.7	7.7
Nonionic, Dobanol 25-7 [®]	3.3	3.3	3.3	3.3	3.3	3.3	3.3	3.3	3.3	3.3
7 NaOH, to pH -	0.86	0.86	0.86	0.86	0.86	0.86	0.86	0.86	0.86	0.86
Na-citrate	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Deflocculating polymer (Narlex DC1 [®])	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Water	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%
Glucose % added on top	10	10	20	20	30	30	40	40	50	50
Sheared	No	Yes	No	Yes	No	Yes	No	Yes	No	Yes
Stability	OK	OK	OK	OK	OK	OK	OK	OK	OK	OK
Viscosity (mPa.s at 21 s-1)	not measured	not measured	not measured	not measured	not measured	not measured	not measured	not measured	not measured	not measured
Optical transmissivity (%)	1.4	14.8	2.2	27.8	1.6	30.8	1.9	40.6	1.7	47.8
Dv, 90 (μm)	5.5	1.19	4.8	1.14	not measured	0.91	not measured	1.35	not measured	1.01

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[0148] Examples 20-22 different sugars + Silverson (shear rate ca 50,000 sec⁻¹). This further illustrates the benefits of shearing and also illustrates the benefit of higher mono- and di-saccharide contents in commercial 'glucose' syrups. It also demonstrates that shearing in the absence of sugar addition (partial refractive index matching) is not sufficient.

Ingredient	% by weight (if not otherwise stated)						
	Comparative example	Comparative example	Ex. 20	Comparative example	Ex. 21	Comparative example (#)	Ex. 22 (##)
LAS-acid	7.7	7.7	7.7	7.7	7.7	7.7	7.7
Nonionic, Dobanol 25-7®	3.3	3.3	3.3	3.3	3.3	3.3	3.3
NaOH, to pH - 7	0.86	0.86	0.86	0.86	0.86	0.86	0.86
Na-citrate	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Deflocculating polymer (Narlex DC1)®	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Water	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%
Glucose % added on top	None	50	50				
Fructose % added on top	None			50	50		
'Glucose' syrup % solids added on top	None					50	50
Sheared	Yes	No	Yes	No	Yes	Yes	Yes
Stability	OK	OK	OK	OK	OK	Separation	OK
Viscosity (mPa.s at 21 s-1)	720	670	670	660	650	not measured	not
Optical transmissivity (%)	1.0	1.7	43.0	2.0	10.5	not measured	22.2
Dv,90 (µm)	not measured	4.8	1.01	9.9	1.25	not measured	not measured
# Cerestar 01411® - of the solids content, 3% is monosaccharide (dextrose), 12% disaccharide (maltose) and 85% tri- and poly saccharides							
## Cerestar 01632® - of the solids content, 38% is monosaccharide (dextrose), 37% disaccharide (maltose) and 25% tri- and poly saccharides							

[0149] Examples 23-26 other shearing devices. This demonstrates that the benefits of shearing are not confined to the Silverson.

Ingredient	% by weight (if not otherwise stated)					
	Comparative example	Comparative example	Ex. 23	Ex. 24	Ex. 25	Ex. 26
LAS-acid	7.7	7.7	7.7	7.7	7.7	7.7

(continued)

Ingredient	% by weight (if not otherwise stated)					
	Comparative example	Comparative example	Ex. 23	Ex. 24	Ex. 25	Ex. 26
Nonionic, Dobanol 25-7®	3.3	3.3	3.3	3.3	3.3	3.3
NaOH, to pH ~ 7	0.86	0.86	0.86	0.86	0.86	0.86
Na-citrate	10.0	10.0	10.0	10.0	10.0	10.0
Deflocculating polymer (Narlex DC1®)	1.0	1.0	1.0	1.0	1.0	1.0
Water	to 100%	to 100%	to 100%	to 100%	to 100%	to 100%
Glucose % added on top	20	20	20	4.35	9.1	20
Sheared	No	Yes	Yes	Yes	Yes	Yes
Approx. shear rate	-	5,000 sec ⁻¹	40,000 to 80,000 sec ⁻¹	ca 10 ⁶ sec ⁻¹	ca 10 ⁶ sec ⁻¹	ca 10 ⁶ sec ⁻¹
Stability	Yes	Yes	Yes	Yes	Yes	Yes
Viscosity (mPa.s at 21 s-1)	not measured	not measured	not measured	not measured	not measured	not measured
Optical transmissivity (%)	1.2	2.7	8.3	6.0	9.7	28.0
Dv, 90 (μm)	4.37	not measured	1.32	not measured	not measured	not measured

- Device in Example 23 is an example of the dynamic mixer described in patent application WO 96/20270. This is a cavity transfer mixer modified by axial movement of the rotor with respect to the stator. This has the function of allowing combination of significant shear and extensional flow fields by arranging the cavities such that the cross-sectional area for flow of the liquid successively increases and decreases by a factor of at least 5 through the apparatus. Results for CI represent averages of different machine operating conditions.
- Examples 24-26 are examples generated via a static shearing device. This is the Model "A" Sonolator, manufactured by Sonic Corporation. Compositions were pumped at backpressures ca 150 bar through a circular nozzle (orifice) of diameter 0.032 cm² at 30°C.
- The second comparative example shows lower level of shear providing insufficient increase in transmittivity.

Example 27: pourable gel.

[0150]

Ingredient	% by weight (if not otherwise stated)	
	Comparative example	Ex. 27
LAS-acid	14.8	14.8
Neodol 1-5 ®	22.23	22.23
NaOH	6.6	6.6
KOH	3.4	3.4

(continued)

Ingredient	% by weight (if not otherwise stated)	
	Comparative example	Ex. 27
to pH	~ 8.4	~ 8.4
Citric acid .laq	13.1	13.1
Deflocculating polymer (Narlex DC1)	4.0	4.0
Water	to 100%	to 100%
Fructose % added on top	0	60
Stability	Yes	Yes
Viscosity (mPa.s at 21 s-1)	450	4730
Optical transmissivity (%)	0.06	5.5

[0151] The foregoing description and Examples illustrate selected embodiments of the present invention. In light thereof, various modifications will be suggested to one skilled in the art.

Claims

1. An aqueous liquid detergent composition having a physical form selected from the group consisting of liquids, pourable gels and non-pourable gels, said composition comprising surfactant, electrolyte and water, which composition is structured with a lamellar phase formed of at least some of the surfactant and at least some of the water, the lamellar structure comprising lamellar droplets, the lamellar droplets being dispersed in an aqueous continuous phase, **characterised in that** the $D_{v,90}$ of the lamellar droplets is less than 2 μm , the composition being substantially clear at 25°C and having an optical transmissivity of at least 5% through a path length of 1cm at 25°C, and wherein the refractive index of the aqueous phase is increased by a sugar dissolved therein.
2. The composition as claimed in Claim 1 **characterised in that** the $D_{v,90}$ of the lamellar droplets is less than 1 μm .
3. The composition of Claim 1 **characterised in that** the difference between the refractive index of the lamellar phase and the refractive index of the aqueous phase is such that the composition has an optical transmissivity of at least 5%.
4. The composition of any preceding Claim **characterised in that** the refractive index of the lamellar phase is decreased by virtue of the surfactant being substantially free of aralkyl surfactant.
5. The composition of any preceding Claim **characterised in** further comprising deflocculating polymer.
6. The composition of Claim 5 **characterised in that** the composition without the deflocculating polymer is colloidally stable.
7. The composition of Claim 6 having a physical form comprising a dispersion of lamellar droplets in an aqueous continuous phase, the composition further comprising deflocculating polymer which composition at 25°C in the absence of the deflocculating polymer does not have a substantially higher viscosity and is colloidally stable.
8. The composition of any preceding Claim **characterised in that** the electrolyte is dissolved in the water.
9. The composition of any preceding Claim **characterised in** having an optical transmissivity of at least 10% through a path length of 1cm at 25°C.
10. The composition of Claim 9 having an optical transmissivity of at least 50% through a path length of 1cm at 25°C.
11. A process for the preparation of the composition of any preceding claim said process **characterised in** comprising

the steps of mixing at least some of the components of the composition at a shear rate of at least $10,000 \text{ s}^{-1}$, and then admixing the resultant composition with any remaining components.

5 Patentansprüche

1. Wässrige, flüssige Waschmittelzusammensetzung mit einer physikalischen Form, ausgewählt aus der Gruppe, bestehend aus Flüssigkeiten, gießbaren Gelen und nichtgießbaren Gelen, wobei die Zusammensetzung Tensid, Elektrolyt und Wasser umfasst, wobei die Zusammensetzung mit einer lamellaren Phase, gebildet aus mindestens etwas von dem Tensid und mindestens etwas von dem Wasser, strukturiert ist, wobei die lamellare Struktur lamellare Tröpfchen umfasst, wobei die lamellaren Tröpfchen in einer wässrigen kontinuierlichen Phase dispergiert sind, **dadurch gekennzeichnet, dass** der $D_{v,90}$ der lamellaren Tröpfchen weniger als $2 \mu\text{m}$ ist, wobei die Zusammensetzung bei 25°C im Wesentlichen klar ist und eine optische Durchlässigkeit von mindestens 5 % über eine Weglänge von 1 cm bei 25°C aufweist, und wobei der Brechungsindex der wässrigen Phase durch einen darin gelösten Zucker erhöht ist.
2. Zusammensetzung nach Anspruch 1, **dadurch gekennzeichnet, dass** der $D_{v,90}$ der lamellaren Tröpfchen weniger als $1 \mu\text{m}$ ist.
3. Zusammensetzung nach Anspruch 1, **dadurch gekennzeichnet, dass** der Unterschied zwischen dem Brechungsindex der lamellaren Phase und dem Brechungsindex der wässrigen Phase derart ist, dass die Zusammensetzung eine optische Durchlässigkeit von mindestens 5 % aufweist.
4. Zusammensetzung nach einem vorangehenden Anspruch, **dadurch gekennzeichnet, dass** der Brechungsindex der lamellaren Phase aufgrund dessen vermindert ist, dass das Tensid im Wesentlichen frei von Aralkyltensid ist.
5. Zusammensetzung nach einem vorangehenden Anspruch, die weiterhin **dadurch gekennzeichnet ist, dass** sie Entflockungspolymer umfasst.
6. Zusammensetzung nach Anspruch 5, **dadurch gekennzeichnet, dass** die Zusammensetzung ohne das Entflockungspolymer kolloidal stabil ist.
7. Zusammensetzung nach Anspruch 6 mit einer physikalischen Form, umfassend eine Dispersion von lamellaren Tröpfchen in einer wässrigen kontinuierlichen Phase, wobei die Zusammensetzung weiterhin Entflockungspolymer umfasst, wobei die Zusammensetzung bei 25°C in Abwesenheit des Entflockungspolymer im Wesentlichen keine höhere Viskosität aufweist und kolloidal stabil ist.
8. Zusammensetzung nach einem vorangehenden Anspruch, **dadurch gekennzeichnet, dass** das Elektrolyt in dem Wasser gelöst ist.
9. Zusammensetzung nach einem vorangehenden Anspruch, **dadurch gekennzeichnet, dass** sie eine optische Durchlässigkeit von mindestens 10 % über eine Weglänge von 1 cm bei 25°C aufweist.
10. Zusammensetzung nach Anspruch 9 mit einer optischen Durchlässigkeit von mindestens 50 % über eine Weglänge von 1 cm bei 25°C .
11. Verfahren zur Herstellung der Zusammensetzung nach einem vorangehenden Anspruch, wobei das Verfahren **dadurch gekennzeichnet ist, dass** es die Schritte des Vermischens von mindestens etwas der Komponenten der Zusammensetzung bei einer Scherrate von mindestens 10000 s^{-1} und dann Anmischen der erhaltenen Zusammensetzung mit beliebigen übrigen Komponenten umfasst.

Revendications

1. Composition détergente liquide aqueuse ayant une forme physique choisie dans le groupe consistant en des liquides, des gels versables et des gels non versables, ladite composition comprenant un électrolyte tensioactif et de l'eau, ladite composition étant structurée avec une phase lamellaire formée d'au moins une partie de l'agent tensioactif et au moins une partie de l'eau, la structure lamellaire comprenant des gouttelettes lamellaires, les gouttelettes

lamellaires étant dispersées dans une phase continue aqueuse, **caractérisée en ce que**

le $D_{v,90}$ des gouttelettes lamellaires est inférieur à $2\text{ }\mu\text{m}$, la composition étant sensiblement limpide à 25°C et ayant une transmissivité optique d'au moins 5 % à travers une distance optique de 1 cm à 25°C . et dans laquelle l'indice de réfraction de la phase aqueuse est accru par un sucre dissous dans celle-ci.

2. Composition selon la revendication 1, **caractérisée en ce que** le $D_{v,90}$ des gouttelettes lamellaires est inférieur à $1\text{ }\mu\text{m}$.
3. Composition selon la revendication 1 **caractérisée en ce que** la différence entre l'indice de réfraction de la phase lamellaire et l'indice de réfraction de la phase aqueuse est telle que la composition ait une transmissivité optique d'au moins 5 %.
4. Composition selon l'une quelconque des revendications précédentes **caractérisée en ce que** l'indice de réfraction de la phase lamellaire est diminué grâce au fait que l'agent tensioactif est sensiblement exempt d'agent tensioactif aralkyle.
5. Composition selon l'une quelconque des revendications précédentes **caractérisée en ce qu'elle** comprend de plus un polymère défloculant.
6. Composition selon la revendication 5 **caractérisée en ce que** la composition sans polymère défloculant est stable sur le plan colloïdal.
7. Composition selon la revendication 6 ayant une forme physique comprenant une dispersion de gouttelettes lamellaires dans une phase continue aqueuse, la composition comprenant également un polymère défloculant, ladite composition à 25°C en l'absence du polymère défloculant n'a pas une viscosité sensiblement plus élevée et est stable sur le plan colloïdal.
8. Composition selon l'une quelconque des revendications précédentes **caractérisée en ce que** l'électrolyte est dissous dans l'eau.
9. Composition selon l'une quelconque des revendications précédentes **caractérisée en ce qu'elle** a une transmissivité optique d'au moins 10 % à travers une distance optique de 1 cm à 25°C .
10. Composition selon la revendication 9 ayant une transmissivité optique d'au moins 50 % à travers une distance optique de 1 cm à 25°C .
11. Procédé pour la préparation de la composition selon l'une quelconque des revendications précédentes, ledit procédé étant **caractérisé en ce qu'il** comprend les étapes consistant à mélanger au moins une partie des composants de la composition avec un gradient de cisaillement d'au moins $10\,000\text{ s}^{-1}$, puis mélanger la composition obtenue avec les composants restants.

REFERENCES CITED IN THE DESCRIPTION

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