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Description

The present invention is concerned with aqueous liquid detergent compositions which contain sufficient detergent-active material and, optionally, sufficiently dissolved electrolyte to result in a structure of lamellar droplets dispersed in a continuous aqueous phase. In particular the present invention relates to liquid detergent compositions having improved solid suspending properties.

Lamellar droplets 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.

Such lamellar dispersions are used to endow properties such as consumer-preferred flow behaviour and/or turbid appearance. Many are also capable of suspending particulate solids such as detergency builders or abrasive particles. Examples of such structured liquids 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 droplet are called 'spherulites'.

The presence of lamellar droplets 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.

The 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.

The viscosity and stability of the product depend on the volume fraction which is occupied by the droplets. Generally speaking, when the volume fraction is around 0.6, the droplets are just touching (space-filling). This allows reasonable stability with an acceptable viscosity (say no more than 2.5 Pa.s, preferably no more than 1 Pa.s at a shear rate of 21s^{-1}). This volume fraction also endows useful solid-suspending properties.

A problem in formulating liquid detergent compositions is to prevent the occurrence of flocculation. When flocculation occurs between the lamellar droplets at a given volume fraction, the viscosity of the corresponding product will increase due to the formation of a network throughout the liquid. Flocculation may also lead to instability reflected in phase separation of the product.

Another problem in formulating liquid detergent compositions of the lamellar droplet type, is that sometimes these compositions are not fully capable of stably suspending solid materials, especially when the volume fraction of lamellar droplets is relatively low, say less than 0.6 or less than 0.5.

It has now been found, that liquid detergent compositions having improved solid suspending properties and/or improved stability and/or improved viscosity can be obtained by carefully controlling the droplet size of the lamellar droplets. In particular an increase in solid suspending properties can be observed -especially when the volume fraction of lamellar droplets is relatively low, say less than 0.6 or less than 0.5- if a relatively high fraction of the lamellar droplets consists of small droplets.

It has been suggested in EP 151 884 (Albright and Wilson) to prepare liquid detergent products comprising dispersed lamellar droplets whereby a major proportion of the droplets have a diameter of 0.2 to 1 micrometer.

It has now been found that the solid suspending properties and/or the stability and/or the viscosity of liquid detergent compositions comprising relatively high amounts of lamellar droplets can be favourably influenced by incorporating therein a deflocculating polymer.

Accordingly the present invention relates to a liquid detergent composition comprising a dispersion of lamellar droplets of detergent active materials in an aqueous continuous phase, said composition also comprising a deflocculating polymer and suspended particles of solid material, wherein at least one of the following conditions are fulfilled:

(1) at least 50 % of the lamellar droplets have a diameter of less than 0.45 micrometer;

(2) the detergent composition has a refractive index for light having a wavelength of 589 nm which is at least 0.01 above the refractive index of its corresponding aqueous continuous phase.

Compositions of the invention may satisfy condition (1), (2) or both conditions as specified above. Both conditions are believed to correspond to liquid compositions, comprising relatively high numbers of small particles.

The percentage of lamellar droplets having a diameter of less than 0.45 micrometer can 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 having a diameter of less than 0.45 micrometer.

Preferably at least 50 % of the lamellar droplets have a diameter of less than 0.35 micrometer, more pre-

ferred less than 0.25 micrometer, most preferred less than 0.15 micrometer, especially preferred less than 0.10 or 0.07 micrometer.

The refractive index of the liquid detergent composition 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 a Atago digital refractometer RX-1000. The use of internal reflection measurements is especially advantageous for determining the refractive index for opaque systems.

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.

Applicants believe that a liquid detergent composition comprising relatively low levels of small particles will generally have a lower refractive index than a corresponding composition, wherein relatively high levels of small particles are present. The reason for this may be the following: in measuring the refractive index of a system, droplets being significantly larger than the wavelength of the measuring light will cause the scattering of incident light, but will not contribute to its refractive power. Calculations using the Mie scattering theory confirm this.

Therefore, theoretically, an aqueous composition, which only comprises lamellar droplets having a particle size significantly above the wavelength of the light, will have a refractive index which is close to the refractive index of the aqueous base of the product. Detergent compositions comprising particles having a size comparable or smaller than the wavelength of the light, will be optically more dense than compositions without these particles. Therefore, for a given composition, an increase in refractive index is a sign of the presence of relatively small particles in the product.

Preferably the refractive index of the total composition is more than 0.02 above the refractive index of the corresponding aqueous phase, more preferred from 0.03 to 0.20, most preferred from 0.04 to 0.15 especially preferred between 0.05 and 0.10.

Compositions of the invention can be obtained by any method for preparing liquid detergent compositions. The man skilled in the art will be able to select the components and their levels in order to allow the formation of a lamellar droplet structure. Also the skilled man will be able to adapt the formulation and/or the processing conditions thereof, such that relatively high levels of small droplets are made.

A particularly advantageous method of preparing relative high levels of small lamellar droplets is the use of high shear in preparing the compositions of the invention. This can for example be accomplished by using a high shear device in a recirculation loop during preparation of the liquid or by applying high shear after the preparation of the liquid. Preferred shear rates are more than 1,000 s⁻¹, more preferred from 2,000 to 150,000, especially preferred from 4,000 to 15,000. These shear conditions are especially suitable for separate recirculation loops. For high shear mixing, generally the average shear will be more than 10, for example from 15 to 200 s⁻¹, more preferred 20 to 100 s⁻¹.

Accordingly the present invention also relates to a liquid detergent composition comprising a dispersion of lamellar droplets of detergent active materials in an aqueous continuous phase, said composition comprising a deflocculating polymer and said composition being obtainable by a process comprising the step of mixing the detergent active materials in water (optionally in the presence of other ingredients of the composition) at a relatively high shear rate and/or by applying relatively high shear to the finished composition.

Preferably compositions obtainable by this method comprise suspended particles of solid material. In the context of the present invention the term suspended solid material refers to any solid material that is not -completely- soluble in the composition. Examples of materials that are usually present in the form of suspended solids are fluorescers, (partially) insoluble builder materials such as STP or zeolites etc, silicon antifoam materials, bleach particles such as perborate bleaches and softener particles. Preferably the level of suspended solid materials is from 0.01 to 50 %, most preferred 1 to 40 % by weight.

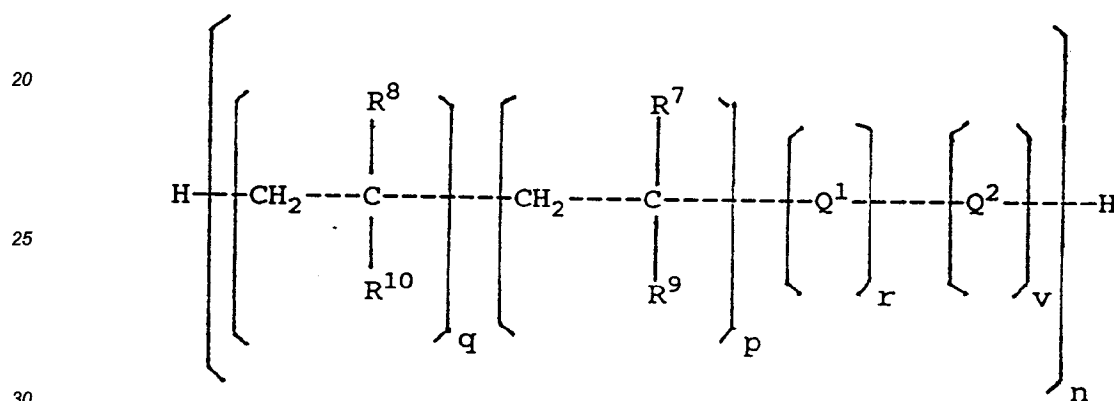
It has been found that the stability and/or the viscosity and/or the solid suspending properties of compositions of the invention can favourably be influenced by incorporating therein a deflocculating polymer. The incorporation of deflocculating polymers is especially useful for stabilising lamellar liquid detergent compositions, wherein the phase volume of the lamellar droplets is relatively low, say less than 0.55 or even less than 0.50 or 0.49. Especially advantageous is the use of deflocculating polymers in compositions having a lamellar phase volume of from 0.40 to 0.48.

Suitable deflocculating polymers for use in compositions of the present invention are for instance described in our copending European patent application 89201530.6 (EP 346 995), polymers as described in this

patent have a hydrophilic backbone and at least one hydrophobic side chain. Generally the hydrophilic backbone of the polymer is predominantly linear (the main chain of the backbone constitutes at least 50 %, preferably more than 75 %, most preferred more than 90% by weight of the backbone), suitable monomer constituents of the hydrophilic backbone are for example unsaturated C₁₋₆ acids, ethers, alcohols, aldehydes, ketones or esters, sugar units, alkoxy units, maleic anhydride and saturated polyalcohols such as glycerol. Examples of suitable monomer units are acrylic acid, methacrylic acid, maleic acid, vinyl acetic acid, glucosides, ethylene oxide and glycerol. The hydrophilic backbone made from the backbone constituents in the absence of hydrophobic side-groups is relatively water-soluble at ambient temperature and a pH of between 6.0 and 14.0. Preferably the solubility is more than 1g/l, more preferred more than 5 g/l most preferred more than 10 g/l.

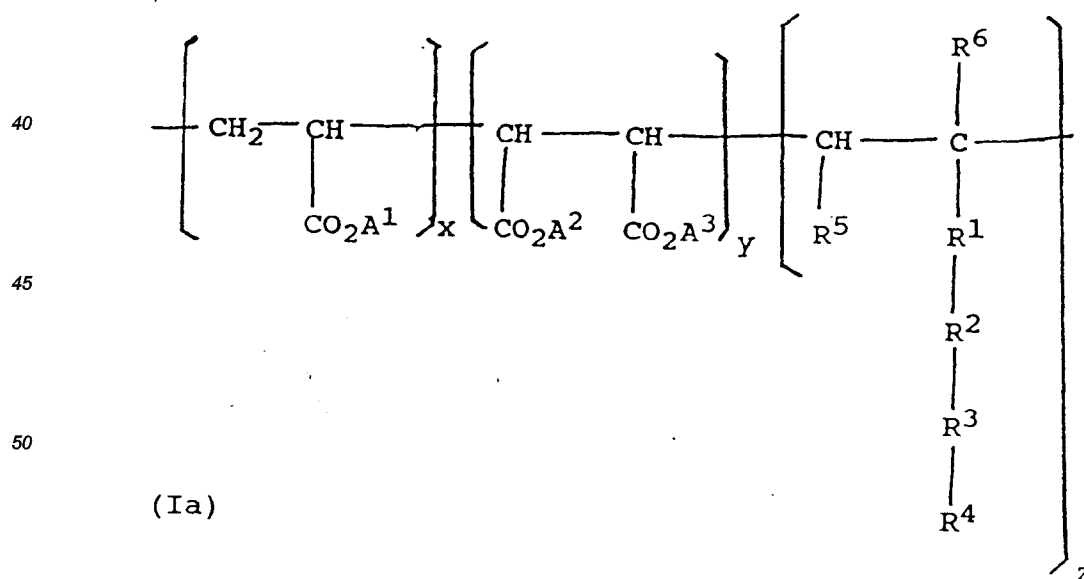
Preferably the hydrophobic sidegroups are composed of relatively hydrophobic alkoxy groups for example butylene oxide and/or propylene oxide and/or alkyl or alkenyl chains having from 5 to 24 carbon atoms. The hydrophobic groups may be connected to the hydrophilic backbone via relatively hydrophilic bonds for example a poly ethoxy linkage.

Preferred polymers are of formula (I):



wherein:

Q² is a molecular entity of formula (Ia):



wherein:

R¹ represents -CO-O-, -O-, -O-CO-, -CH₂-, -CO-NH- or is absent;

R² represents from 1 to 50 independently selected alkyleneoxy groups preferably ethylene oxide or propylene oxide groups, or is absent, provided that when R³ is absent and R⁴ represents hydrogen or contains no more

than 4 carbon atoms, then R^2 must contain an alkyleneoxy group preferably more than 5 alkyleneoxy groups with at least 3 carbon atoms;

R^3 represents a phenylene linkage, or is absent;

5 R^4 represents hydrogen or a C_{1-24} alkyl or C_{2-24} alkenyl group, with the provisos that

a) when R^1 represents $-O-CO-$, R^2 and R^3 must be absent and R^4 must contain at least 5 carbon atoms;

b) when R^2 is absent, R^4 is not hydrogen and when also R^3 is absent, then R^4 must contain at least 5 carbon atoms;

R^5 represents hydrogen or a group of formula $-COOA^4$;

10 R^6 represents hydrogen or C_{1-4} alkyl; and

A^1, A^2, A^3 and A^4 are independently selected from hydrogen, alkali metals, alkaline earth metals, ammonium and amine bases and C_{1-4} , or $(C_2H_4O)_tH$ wherein t is from 1-50, and wherein the monomer units may be in random order.

15 Q^1 is a multifunctional monomer, allowing the branching of the polymer, wherein the monomers of the polymer may be connected to Q^1 in any direction, in any order, therewith possibly resulting in a branched polymer. Preferably Q^1 is trimethyl propane triacrylate (TMPTA), methylene bisacrylamide or divinyl glycol.

n is at least 1; z and v are 1; and $(x + y + p + q + r) : z$ is from 4 : 1 to 1,000 : 1, preferably from 6 : 1 to 250 : 1; in which the monomer units may be in random order; and preferably either p and q are zero, or r is zero; especially preferably p, q, y and r are zero.

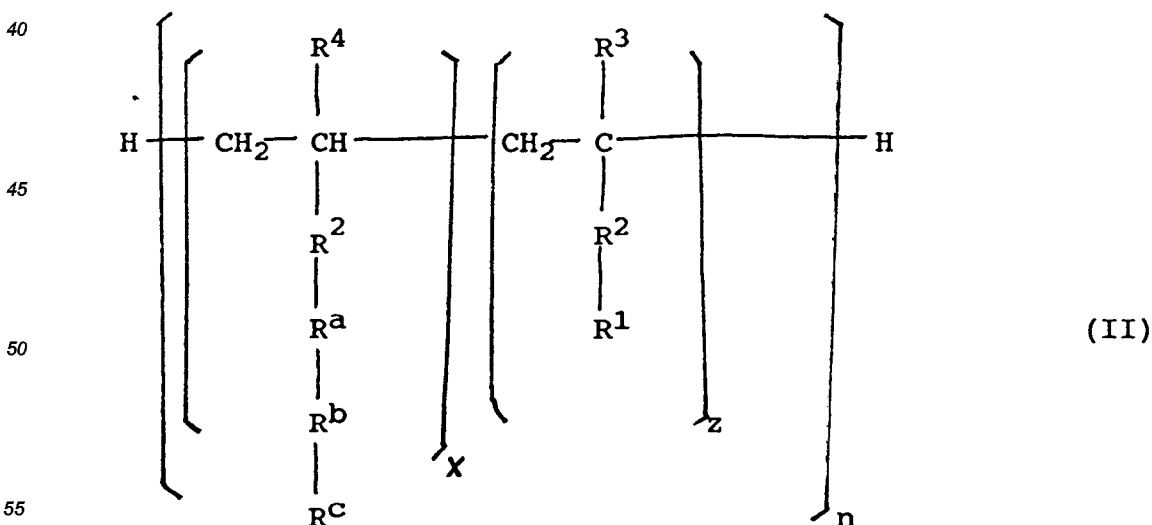
20 R^7 and R^8 represent $-CH_3$ or $-H$;

R^9 and R^{10} represent substituent groups such as amino, amine, amide, sulphonate, sulphate, phosphonate, phosphate, hydroxy, carboxyl and oxide groups, preferably they are selected from $-SO_3Na$, $-CO-O-C_2H_4-OSO_3Na$, $-CO-O-NH-C(CH_3)_2-SO_3Na$, $-CO-NH_2$, $-O-CO-CH_3$, $-OH$;

25 Preferably polymers for use in compositions of the invention which are of relatively high pH (say 10 or more) are substantially free of hydrolysable groups such as carbonyl groups for increased polymer stability at high pH values. Particularly preferred polymers for use in high pH compositions of the invention comprise hydrophilic backbones constituted by acid groups such as acrylic acid and at least one hydrophobic side chain which is constituted of from 5 to 75 relatively water-insoluble alkoxy groups such as propoxy units optionally linked to the hydrophilic backbone via an poly-alkoxy linkage constituted of from 1-10 relatively watersoluble alkoxy groups such as ethoxy units.

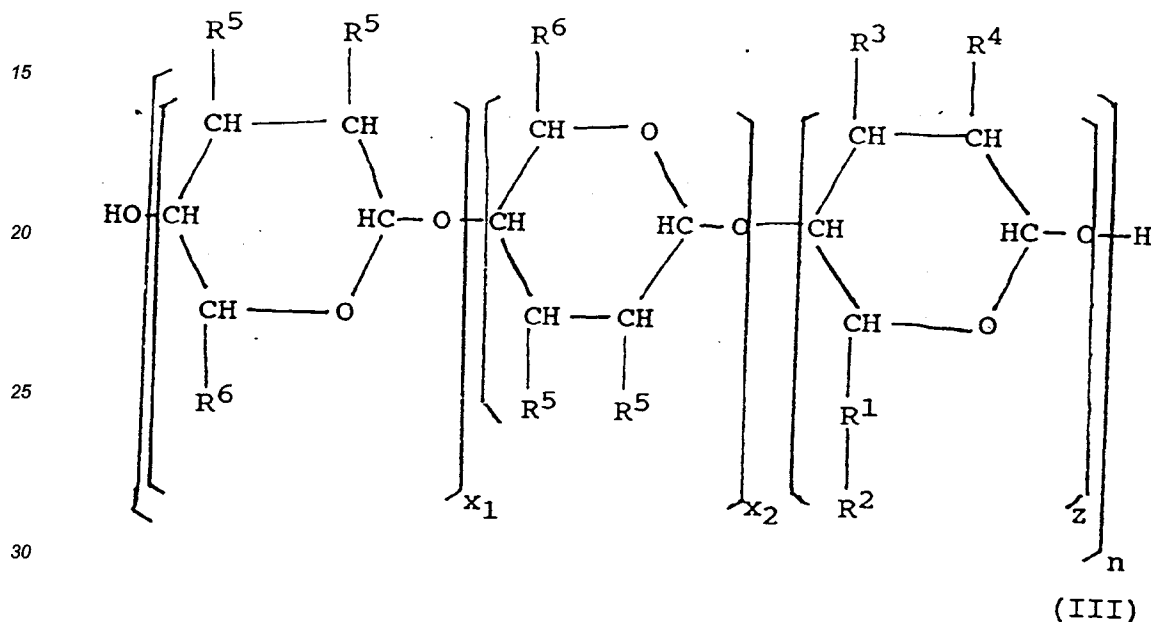
30 Other preferred polymers for use in compositions of the invention are described in our copending non-published patent applications WO/91/06622 (published on 16 May 1991), WO/91/06623 (published on 16 May 1991) and GB 2,237,813 (published on 16 May 1991). Of the polymers described in those patent applications, especially the use of polymers in accordance with WO/91/06623 is preferred. These polymers are constituted of nonionic monomers and ionic monomers, wherein the ionic monomer is from 0.1 to 50 % by weight of the polymer.

Especially preferred polymers of this type are of the formula:



wherein: x, z and n are as above;

- R³ and R⁴ represent hydrogen or C₁₋₄ alkyl;
 - R² represents -CO-O-, -O-, -O-CO-, -CH₂-, -CO-NH-, or is absent;
 - R¹ represents -C₃H₆-N⁺-(CH₃)₃Cl⁻, -C₂H₄-OSO₃⁻(Na⁺), -SO₃⁻(Na⁺), -C₂H₄ N⁺(CH₃)₃ Cl⁻, -C₂H₄ N⁺(C₂H₅)₃ Cl⁻, -CH₂ N⁺(CH₃)₃ Cl⁻, -CH₂ N⁺(C₂H₅)₃ Cl⁻ or benzyl-SO₃⁻ Na⁺;
 - R^a is CH₂, C₂H₄, C₃H₆ or is absent;
 - R^b represents from 1 to 50 independently selected alkylene oxide groups, preferably ethylene oxide groups or is absent;
 - R^c represents -OH or -H;
- and wherein if R², R^a and R^b are absent, then R^c is not -H.
- Other preferred polymers have the formula:



Wherein

- $x = x_1 + x_2$
- x, z and n are as defined above
- R¹ represents -CH₂O- or -O-;
- R² represents -CH₂COO⁻Na⁺, -C₃H₆N⁺(CH₃)₃Cl⁻ or -C₃H₆ON⁺(CH₃)₃Cl⁻
- R³ and R⁴ represents -OH, CH₂OH, -O(C₃H₆O)_p-H, -CH₂-O(C₃H₆O)_p-H or -OCH₂COO⁻Na⁺, -O-C₃H₆ON⁺(CH₃)₃Cl⁻ or -O-C₃H₆N⁺(CH₃)₃Cl⁻
- R⁵ represents -OH, -NH-CO-CH₃ or -O(C₃H₆O)_p-H
- R⁶ represents -OH, -CH₂OH, -CH₂-OCH₃, -O(C₃H₆O)_p-H or -CH₂-O-(C₃H₆O)_p-H
- p is from 1 - 10.

Preferably polymers for use in compositions have a molecular weight (as determined as in our co-pending patent application EP 346,995) of between 500 and 100,000, more preferred from 1,000 to 50,000, especially preferred from 2,000 to 20,000 most preferred from 4,000 to 15,000. Polymers for use in compositions of the invention may for example be prepared by using conventional aqueous polymerisation procedures, suitable methods are for example described in the above mentioned co-pending european patent application.

Generally the defloculating polymer will be used at from 0.01 to 5 % by weight of the composition, more preferably from 0.1 to 3.0, especially preferred from 0.25 to 3.0 %, most preferred from 0.5 to 2.5 %.

Without being bound by any particular interpretation or theory, the Applicants have hypothesized that the polymers exert their action on the composition by the following mechanism. The hydrophobic side-chain(s) or ionic groups could be incorporated in or onto the outer bi-layer of the droplets, leaving the hydrophilic or nonionic backbone over the outside of the droplets and/or the polymers could be incorporated deeper inside the droplet.

When the hydrophobic or side chains or ionic groups are incorporated in or onto the outer bilayer of the droplets, this has the effect of decoupling the inter-and intra-droplet forces i.e. the difference between the forces between individual surfactant molecules in adjacent layers within a particular droplet and those be-

tween surfactant molecules in adjacent droplets could become accentuated in that the attractive forces between adjacent droplets are reduced. This will generally result in an increased stability due to less flocculation and a decrease in viscosity due to smaller attractive forces between the droplets resulting in greater distances between adjacent droplets.

The polymers can also be incorporated deeper inside the droplets, then possibly also less flocculation will occur, resulting in an increase in stability. The influence of these polymers within the droplets on the viscosity is governed by two opposite effects : firstly the presence of deflocculating polymers will decrease the attractive forces between adjacent droplets resulting in greater distances between the droplets, generally resulting in a lower viscosity of the system; secondly the attractive forces between the layers within the droplets are equally reduced by the presence of the polymers in the droplet, this generally result in an increase in the layer thickness, therewith increasing the lamellar volume of the droplets, therewith increasing the viscosity. The net effect of these two opposite effects may result in either a decrease or an increase in the viscosity of the product.

Preferred compositions according to the invention are physically stable and have a relatively low viscosity. Preferably a corresponding composition minus the deflocculating polymer is less stable and/or has a higher viscosity.

In the context of the present invention, physical stability for these systems can be defined in terms of the maximum separation compatible with most manufacturing and retail requirements. That is, the 'stable' compositions will yield no more 10 %, preferably no more than 5 %, most preferred no more than 2% by volume phase separation as evidenced by appearance of 2 or more separate phases when stored at 25°C for 21 days from the time of preparation.

Preferably, compositions of the invention have a pH between 6 and 14, more preferred from 6.5 to 13, especially preferred from 7 to 12.

Compositions of the invention preferably have a viscosity of less than 2,500 mPa.s at 21 s⁻¹, more preferred less than 1,500 mPa.s, most preferred less than 1,000 mPa.s, especially preferred between 100 and 750 mPa.s at 21 s⁻¹. Also preferably the viscosity at a shear rate of 10⁻⁴ s⁻¹ is at least 10,000 mPa.s, more preferred more than 100,000, especially preferred more than 1,000,000, most preferred more than 10,000,000 mPa.s.

Compositions of the invention also comprise detergent active materials, preferably at a level of from 1 to 70% by weight of the composition, more preferred a level of 5 to 40 % by weight, most preferred from 10 to 35 % by weight.

In the case of blends of surfactants, the precise proportions of each component which will result in lamellar structures will depend on the type(s) and amount(s) of the electrolytes, as is the case with conventional structured liquids.

In the widest definition the detergent-active material 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.I, 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.

Suitable nonionic surfactants include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, amides or alkyl phenols 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 sulfoxides.

Preferably the level of nonionic surfactant materials is from 1 -40 % by weight of the composition, more preferred from 2-20 %.

Compositions of the present invention may contain synthetic anionic surfactant ingredients, which are preferably present in combination with the above mentioned nonionic materials. Suitable anionic surfactants are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher (C₈-C₁₈) alcohols produced, for example, from tallow or coconut oil, sodium and potassium alkyl (C₉-C₂₀) benzene sulphonates, particularly sodium linear secondary

alkyl (C₁₀-C₁₅) 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; sodium coconut oil fatty monoglyceride sulphates and sulphonates; sodium and potassium salts of sulphuric acid esters of higher (C₈-C₁₈) 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 neutralized with sodium hydroxide; sodium and potassium salts of fatty acid amides of methyl taurine; alkane monosulphonates such as those derived by reacting alpha-olefins (C₈-C₂₀) with sodium bisulphite and those derived from reacting paraffins with SO₂ and Cl₂ 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 C₁₀-C₂₀ alpha-olefins, with SO₃ and then neutralizing and hydrolyzing the reaction product. The preferred anionic detergent compounds are sodium (C₁₁-C₁₆) alkyl benzene sulphonates and sodium (C₁₆-C₁₈) alkyl sulphates.

Generally the level of the above mentioned non-soap anionic surfactant materials is from 1-25 % by weight of the composition, more preferred from 2 to 15 %.

It is also possible, and sometimes preferred, to include an alkali metal soap of a mono- or dicarboxylic acid, especially a soap of an acid having from 12 to 18 carbon atoms, for example oleic acid, ricinoleic acid, alke(ny)l succinate for example dodecyl succinate, and fatty acids derived from castor oil, rapeseed oil, groundnut oil, coconut oil, palmkernel oil or mixtures thereof. The sodium or potassium soaps of these acids can be used. Preferably the level of soap in compositions of the invention is from 1-35 % by weight of the composition, more preferred from 5-25 %.

Also possible is the use of salting out resistant active materials for example those described in EP 328 177, especially the use of alkyl poly glycoside surfactants for example those disclosed in EP 70 074.

Also alkyl mono glucosides may be used.

The compositions optionally also contain electrolyte in an amount sufficient to bring about lamellar structuring of the detergent-active material. 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; that is salting-out electrolytes have a lyotropic number of less than 9.5. Optionally, some salting-in electrolyte (as defined in the latter specification) may also be included.

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. In this context it should be noted that some detergent active materials such as for example soaps, also have builder properties.

Examples of phosphorous-containing inorganic detergency builders include the water-soluble salts, especially alkali metal pyrophosphates, orthophosphates, polyphosphates and phosphonates. Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, phosphates and hexametaphosphates. Phosphonate sequestrant builders may also be used. Sometimes it is however preferred to minimise the amount of phosphate builders.

Examples of non-phosphorus-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.

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.

Examples of organic detergency builders, when present, include the alkaline metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates. Specific examples include sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, melitic acid, benzene polycarboxylic acids, CMOS, tartrate mono succinate, tartrate di succinate and citric acid. Citric acids or salts thereof are preferred builder materials for use in compositions of the invention.

In the context of organic builders, it is also desirable to incorporate polymers which are only partly dissolved, in the aqueous continuous phase as described in EP 301.882. This allows a viscosity reduction (due 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. Typical amounts are from 0.5 to 4.5% by weight.

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 100ml of a 5% by weight

aqueous solution of the polymer, said second polymer also having a vapour pressure in 20% aqueous solution, equal to 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 6000; said second polymer having a molecular weight of at least 1000. Use of such polymers is generally described in our EP 301,883. Typical levels are from 0.5 to 4.5% by weight.

Preferably the level of non-soap builder material is from 5-40 % by weight of the composition, more preferred from 5 to 25 % by weight of the composition.

Apart from the ingredients already mentioned, a number of optional ingredients may also be present, for example lather boosters such as alkanolamides, particularly the monoethanolamides derived from palm kernel fatty acids and coconut fatty acids, 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, enzymes such as proteases, amylases and lipases (including Lipolase (Trade Mark) ex Novo), enzyme stabilisers, anti-redeposition agents, germicides and colourants.

Compositions of the invention may be prepared by any conventional method for the preparation of liquid detergent compositions, although the use of high shear conditions is preferred. A preferred method involves the dispersing of the electrolyte ingredient (if present) 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 may for example be added after the electrolyte ingredient or as the final ingredient. It is sometimes preferable that the deflocculating polymers are added prior to the formation of the lamellar structure. Alternatively part of the polymer may be added prior to the formation of the lamellar structure and the remaining part of the polymer is added as the final ingredient. Also it is sometimes preferred to add all or a major part of the polymer as the final ingredient.

In use the detergent compositions of the invention will be diluted with wash water to form a wash liquor for instance for use in a washing machine. The concentration of liquid detergent composition in the wash liquor is preferably from 0.1 to 10 %, more preferred from 0.1 to 3% by weight.

The invention will now be illustrated by way of the following Examples.

EXAMPLE I

For illustrating the correlation between the size of the lamellar droplets and the increase in the refractive index, the following compositions (which are free from suspended solids) were made by mixing the citrate and NaOH (in an amount sufficient to neutralise the LAS-acid) into water of 40 °C, followed by the addition of the deflocculating polymer -if any-. The LAS-acid and the Synperonic A7® are added as a premix as the final ingredients. The samples containing 2 % or 4 % of deflocculating polymer were divided in two parts one of the parts being subjected to high shear conditions (70,000 s⁻¹) using an Ultra Turrax.

Of all the samples the refractive index for 589 nm light was measured using the Atago digital refractometer RX-1000. The lamellar droplet size of the unsheared samples was measured from electron microscopy pictures at a magnification of 15,000 x.

FORMULATION	wt parts	A	B	C	D	E
NaLAS ²⁾		<-----23.3----->				
Synperonic A7 [®]		<-----10.0----->				
Na-citrate 2aq		<-----16.7----->				
Water		<-----50.0----->				
polymer ¹⁾		-	0.5	1.0	2.0	4.0

1) deflocculating polymer of formula A-11 of EP 346 995.

2) derived from Marlon AS3[®]

The following results were obtained:

	sample	δRI ¹⁾		lamellar droplets ²⁾ μm
		no high shear	high shear	
	A	0.000	--	-- ³⁾
	B	0.0106	--	0.12
	C	0.0154	--	0.09
	D	0.0229	0.0360	0.06
	E	0.0338	0.0447	0.04

1) difference between Refractive index (n_D^{25}) and refractive index of isolated continuous aqueous phase.

2) 50 % of the droplets have a diameter of less than the value indicated (no high shear).

3) impossible to measure due to strong flocculation.

These results indicate that the decrease of lamellar droplet size correlates to an increase of refractive index and that high shear conditions can advantageously be used for obtaining a decrease of the lamellar droplet size.

EXAMPLE II

The following compositions were made by mixing the borax and the citrate in water of 50 °C, followed by the addition of the zeolite material and the deflocculating polymer. The LAS and the Synperonic A7® are added as a pre-mix, whereby the LAS is added in acid form. The LAS-acid is neutralised with NaOH. Finally the glycerol is added and the mixture is cooled. Composition B was prepared such that 50 % of the lamellar droplets had a diameter of 0.12 micrometer or less. The number of droplets was determined by using x30,000 electron microscopy pictures.

	INGREDIENT % (wt)	A	B
	NaLAS		18.4
	Synperonic A7®		11.5
	Glycerol		5.5
	Borax		3.8
	Na-citrate		7.7
	Zeolite (Wessalith P)®		16.4
	deflocculating polymer	--	1.0
	water	balance	

polymer A44 as described in EP 89201530.6 (EP 346 995)

Compositions A and B were liquid detergent compositions comprising a dispersion of lamellar droplets. Composition A was a flocculated highly viscous system which was unstable and had poor suspending properties, the zeolite particles were not stably suspended and formed a sediment upon storage at 25 °C. Composition B deflocculated and well pourable and had good suspending properties, the zeolite was stably sus-

pended in the system. This example illustrates that small lamellar droplets in combination with deflocculating polymers can provide increased stability.

5 Example III

The following formulations each were made by two methods: formulations A1-D1 were made by mixing the NaOH, borax, citrate and glycerol in water of 50 °C, followed by the addition of the deflocculating polymer and a premix of the LAS, Fatty acid and the Synperonic A7®. Formulations A2-D2 were of the same composition as A1-D1 except that they were prepared by mixing the Glycerol, Borax, NaOH and citrate in water of 50 °C followed by the addition of the active premix and finally adding the deflocculating polymer.

Formulation (% wt)	A	B	C	D
15 LAS acid (Marlon As3)	<-----10.3----->			
Synperonic A7®	<-----16.0----->			
Fatty acid ²⁾	<-----10.0----->			
20 Na-citrate 2aq	<-----10.0----->			
Glycerol	<-----5.0----->			
Borax	<-----3.5----->			
NaOH	<-----6.0----->			
25 Polymer ¹⁾	0.18	0.36	0.72	1.06
Water	<-----balance----->			

30 1) Polymer A-11 of EP 346 995 (deflocculating polymer of formula I, wherein q, p and r are 0, v=1, x=25, y=0, R¹ is -CO-O-, R² is absent R³ is absent, R⁴ is

35 -C₁₂H₂₅, R⁵ is -H, R⁶ is -CH₃ and A¹ is Na. The molecular weight of the polymer is about 3.5 K).

2) 60/40 mixture of Priolene 6902^② and Prifac 7904^②.

40 For each of the formulations the Delta-RI (difference between refractive index of product and refractive index of continuous phase of composition minus deflocculating polymer) was measured as well as the physical stability upon storage for 21 days at 25 °C.

The following results were obtained:

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	<u>FORMULATION</u>	<u>Delta RI</u>	<u>% phase separation</u>
	A1	0.0179	7 %
5	A2	0.0299	0 %
	B1	0.0164	7 %
	B2	0.0298	0 %
10	C1	0.0179	5 %
	C2	0.0289	0 %
	D1	0.0179	3 %
	D2	0.0289	0 %
15	Comparative ¹⁾	0.001	35 %

1) composition without deflocculating polymer.

This example clearly indicates that an increased stability can be obtained by adding a deflocculating polymer to a formulation. The stability increase is especially pronounced in systems having a high Delta-RI.

Example IV

The following compositions were made as in example II

	<u>INGREDIENT (%wt)</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>
30	NaLas	<-----18.4----->					
	Synperonic A7	<-----11.5----->					
	Na-citrate 2aq	<-----6.7----->					
35	Glycerol	<-----5.5----->					
	Borax	<-----3.8----->					
	Zeolite (WessalithP)	<-----16.4----->					
40	water	<-----37.7----->					
	polymer ¹⁾	--	0.5	1.0	1.5	2.0	2.5

1) polymer A-11 of EP 346 995, percentage on top of the formulation.

The physical properties of the formulation were as follows:

	COMP	viscosity ¹⁾	stability ²⁾	deltaRI ³⁾
5	A	3,550	4)	0.0051
	B	600	5)	not measurable
	C	570	5)	0.0043
10	D	700	5)	0.0050
	E	820	6)	0.0245
	F	1,290	6)	0.0331

- 15 1) viscosity at 21 s⁻¹ at ambient temperature
 2) after storage for 3 weeks at ambient temperature
 3) difference between refractive index of product and
 20 refractive index of isolated aqueous phase
 4) strongly flocculated, highly unstable
 5) partly deflocculated, unstable, zeolite
 sedimentation
 25 6) deflocculated, stable, no zeolite sedimentation

30 These examples illustrate that small lamellar droplets as evidenced by a relatively high deltaRI can provide increased stability and less zeolite sedimentation.

Claims

- 35 1. A liquid detergent composition comprising a dispersion of lamellar droplets of detergent active materials in an aqueous continuous phase, said composition also comprising a deflocculating polymer and suspended particles of solid material, wherein at least one of the following conditions are fulfilled:
 (1) at least 50 % of the lamellar droplets have a diameter of less than 0.45 micrometer;
 (2) the detergent composition has a refractive index for light having a wavelength of 589 nm which is
 40 at least 0.01 above the refractive index of its corresponding aqueous continuous phase.
2. A liquid detergent composition comprising a dispersion of lamellar droplets of detergent active materials in an aqueous continuous phase, said composition comprising a deflocculating polymer and said composition being obtainable by a process comprising the step of mixing the detergent active materials in
 45 water (optionally in the presence of other ingredients of the composition) at a shear rate of more than 1,000 s⁻¹ and/or by applying an average shear of more than 10 s⁻¹ to the finished product.
3. Composition according to claim 2, comprising suspended particles of solid materials.
- 50 4. Composition according to claim 1 or 2, wherein the deflocculating polymer is of the formula I, II or III as specified hereafter :

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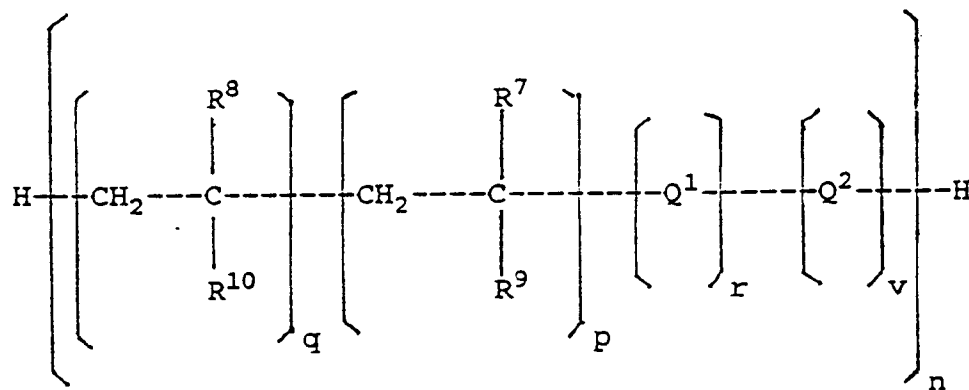
Formula (I):

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wherein:

Q² is a molecular entity of formula (Ia):

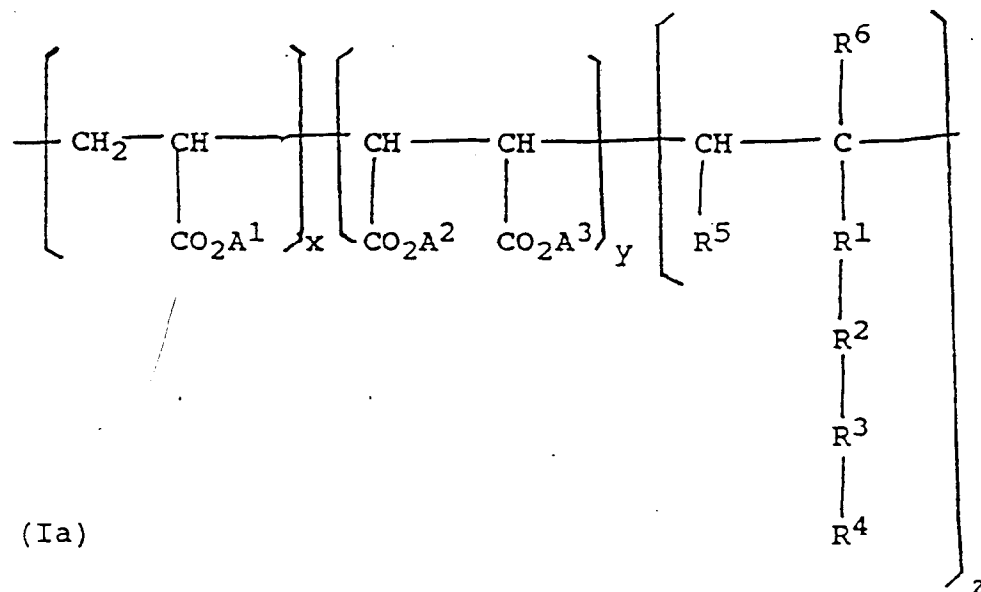
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35

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(Ia)



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wherein:

R¹ represents -CO-O-, -O-, -O-CO-, -CH₂-, -CO-NH- or is absent;R² represents from 1 to 50 independently selected alkyleneoxy groups preferably ethylene oxide or propylene oxide groups, or is absent, provided that when R³ is absent and R⁴ represents hydrogen or contains no more than 4 carbon atoms, then R² must contain an alkyleneoxy group preferably more than 5 alkyleneoxy groups with at least 3 carbon atoms;

50

R³ represents a phenylene linkage, or is absent;R⁴ represents hydrogen or a C₁₋₂₄ alkyl or C₂₋₂₄ alkenyl group, with the provisos thata) when R¹ represents -O-CO-, R² and R³ must be absent and R⁴ must contain at least 5 carbon atoms;

55

b) when R² is absent, R⁴ is not hydrogen and when also R³ is absent, then R⁴ must contain at least 5 carbon atoms;R⁵ represents hydrogen or a group of formula -COOA⁴;R⁶ represents hydrogen or C₁₋₄ alkyl; and

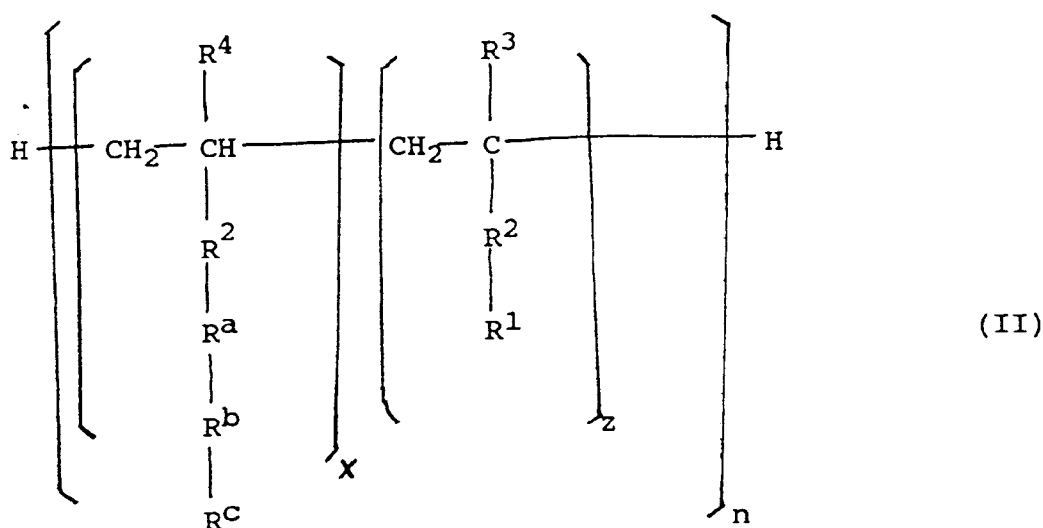
A¹, A², A³ and A⁴ are independently selected from hydrogen, alkali metals, alkaline earth metals, ammonium and amine bases and C₁₋₄, or (C₂H₄O)_tH wherein t is from 1-50, and wherein the monomer units may be in random order.

Q¹ is a multifunctional monomer, allowing the branching of the polymer, wherein the monomers of the polymer may be connected to Q¹ in any direction, in any order, therewith possibly resulting in a branched polymer. Preferably Q¹ is trimethyl propane triacrylate (TMPTA), methylene bisacrylamide or divinyl glycol.

n is at least 1; z and v are 1; and (x + y + p + q + r) : z is from 4 : 1 to 1,000 : 1, preferably from 6 : 1 to 250 : 1; in which the monomer units may be in random order; and preferably either p and q are zero, or r is zero; especially preferably p, q, y and r are zero.

R⁷ and R⁸ represent -CH₃ or -H;

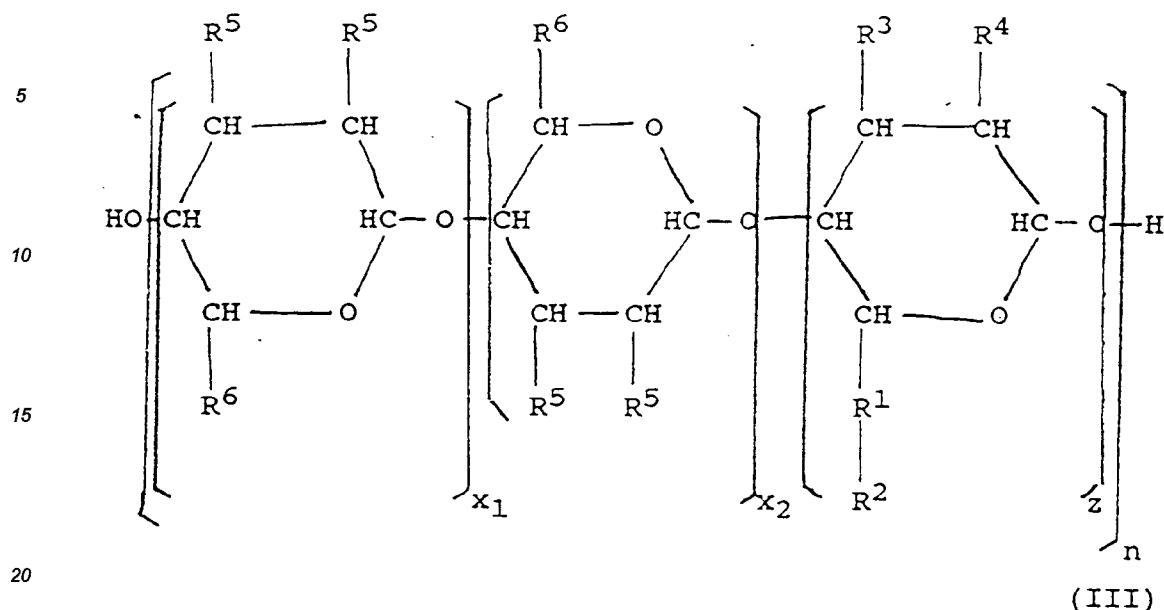
R⁹ and R¹⁰ represent substituent groups such as amino, amine, amide, sulphonate, sulphate, phosphonate, phosphate, hydroxy, carboxyl and oxide groups, preferably they are selected from -SO₃Na, -CO-O-C₂H₄-OSO₃Na, -CO-O-NH-C(CH₃)₂-SO₃Na, -CO-NH₂, -O-CO-CH₃, -OH;



wherein: x, z and n are as above;

- R³ and R⁴ represent hydrogen or C₁₋₄ alkyl;
- R² represents -CO-O-, -O-, -O-CO-, -CH₂-, -CO-NH-, or is absent;
- R¹ represents -C₃H₆-N⁺-(CH₃)₃Cl⁻, -C₂H₄-OSO₃(Na⁺), -SO₃⁻(Na⁺), -C₂H₄ N⁺(CH₃)₃ Cl⁻, -C₂H₄ N⁺(C₂H₆)₃ Cl⁻, -CH₂ N⁺(CH₃)₃ Cl⁻, -CH₂ N⁺(C₂H₆)₃ Cl⁻ or benzyl-SO₃⁻ Na⁺;
- R^a is CH₂, C₂H₄, C₃H₆ or is absent;
- R^b represents from 1 to 50 independently selected alkylene oxide groups, preferably ethylene oxide groups or is absent;
- R^c represents -OH or -H;

and wherein if R², R^a and R^b are absent, then R^c is not -H.



Wherein:

- $x = x_1 + x_2$
 - x, z and n are as defined above
 - R^1 represents $-\text{CH}_2\text{O}-$ or $-\text{O}-$;
 - R^2 represents $-\text{CH}_2\text{COO}^-\text{Na}^+$, $-\text{C}_3\text{H}_6\text{N}^+(\text{CH}_3)_3\text{Cl}^-$ or $-\text{C}_3\text{H}_6\text{ON}^+(\text{CH}_3)_3\text{Cl}^-$
 - R^3 and R^4 represents $-\text{OH}$, CH_2OH , $-\text{O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$, $-\text{CH}_2\text{-O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$ or $-\text{OCH}_2\text{COO}^-\text{Na}^+$, $-\text{O}-\text{C}_3\text{H}_6\text{ON}^+(\text{CH}_3)_3\text{Cl}^-$ or $-\text{O}-\text{C}_3\text{H}_6\text{N}^+(\text{CH}_3)_3\text{Cl}^-$
 - R^5 represents $-\text{OH}$, $-\text{NH}-\text{CO}-\text{CH}_3$ or $-\text{O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$
 - R^6 represents $-\text{OH}$, $-\text{CH}_2\text{OH}$, $-\text{CH}_2\text{-OCH}_3$, $-\text{O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$ or $-\text{CH}_2\text{-O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$
 - p is from 1 - 10.
5. Composition according to claim 1 or 2 having a lamellar phase volume of less than 0.55.
 6. Composition according to claim 1 or 2 yielding less than 10 % by volume phase separation as evidences by appearance of 2 or more separate phases when stored at 25 °C for 21 days from the time of preparation.
 7. Composition according to claim 1 or 2 having a viscosity at 21 s⁻¹ of less than 2,500 mPa.s.
 8. Composition according to claim 1 or 2 comprising 1-70 % by weight of detergent active materials, 1-60 % by weight of salting out electrolytes and 0.01 to 5 % by weight of deflocculating polymers.

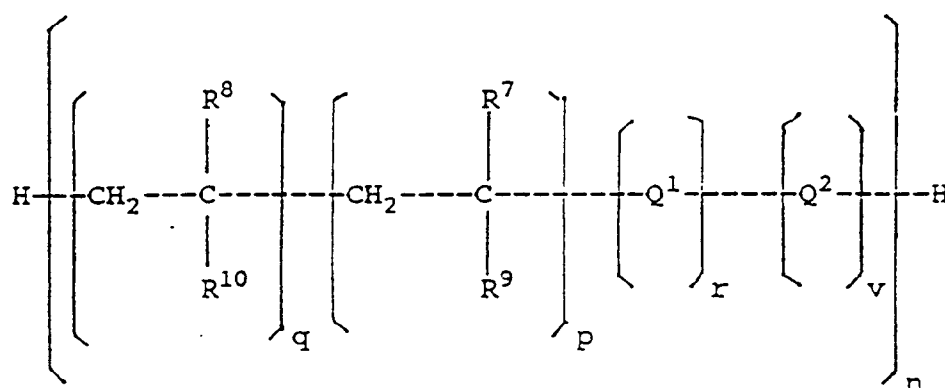
Patentansprüche

1. Flüssige Reinigungsmittelzusammensetzung, umfassend eine Dispersion lamellarer Tröpfchen reinigungsmittelaktiver Materialien in einer wäßrigen kontinuierlichen Phase, wobei die Zusammensetzung ferner ein entfloccendes Polymer und suspendierte Teilchen festen Materials umfaßt und mindestens eine der folgenden Bedingungen erfüllt ist:
 1. mindestens 50% der lamellaren Tröpfchen weisen einen Durchmesser von weniger als 0,45 µm auf;
 2. die Reinigungsmittelzusammensetzung weist ein Lichtbrechungsindex bei einer Wellenlänge von 589 nm auf, der um mindestens 0,01 über dem Brechungsindex ihrer entsprechenden wäßrigen kontinuierlichen Phase liegt.
2. Flüssige Reinigungsmittelzusammensetzung mit einer Dispersion lamellarer Tröpfchen reinigungsmittelaktiver Materialien in einer wäßrigen kontinuierlichen Phase, wobei die Zusammensetzung ein entfloccendes Polymer umfaßt und im Rahmen eines Verfahrens, das die Stufe eines Vermischens der rei-

nigungsmittelaktiven Materialien in Wasser (gegebenenfalls in Anwesenheit weiterer Bestandteile der Zusammensetzung) bei einer Scherrate von mehr als 1000 s^{-1} umfaßt, und/oder durch Ausüben einer durchschnittlichen Scherkraft von mehr als 10 s^{-1} auf das Fertigprodukt erhältlich ist.

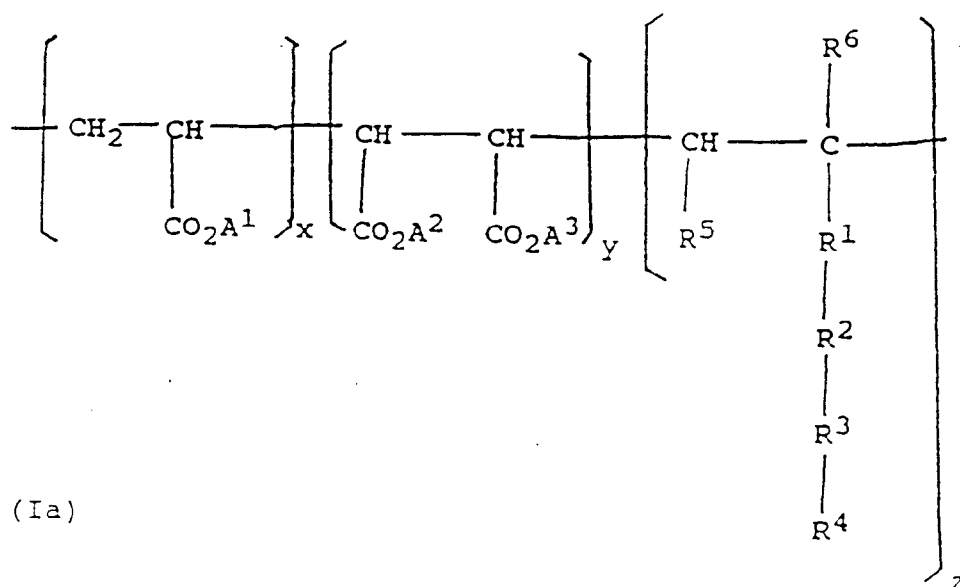
3. Zusammensetzung nach Anspruch 2, umfassend suspendierte Teilchen fester Materialien.
4. Zusammensetzung nach Anspruch 1 oder 2, wobei das entflokkende Polymer einer der folgenden Formeln I, II oder III entspricht:

Formel I:



worin bedeuten:

Q^2 eine Moleküleinheit der Formel Ia:



worin R^1 für $-\text{CO}-\text{O}-$, $-\text{O}-$, $-\text{O}-\text{CO}-$, $-\text{CH}_2-$, $-\text{CO}-\text{NH}-$ steht oder fehlt,

R^2 eine bis 50 unabhängig voneinander ausgewählte Alkylendioxygruppen, vorzugsweise Ethylendioxy- oder Propylendioxygruppen darstellt oder fehlt, vorausgesetzt, daß, wenn R^3 fehlt und R^4 für Wasserstoff steht oder nicht mehr als 4 Kohlenstoffatome enthält, R^2 eine Alkylendioxygruppe, vorzugsweise mehr als 5 Alkylendioxygruppen mit mindestens 3 Kohlenstoffatomen, enthalten muß,

R^3 eine Phenylverbindung darstellt oder fehlt,

R^4 Wasserstoff oder eine C_1 - C_{24} Alkyl- oder C_2 - C_{24} Alkenylgruppe bedeutet, wobei gilt, daß

a) wenn R^1 für -O-CO- steht, R^2 und R^3 fehlen müssen und R^4 mindestens 5 Kohlenstoffatome enthalten muß,

b) wenn R^2 fehlt, R^4 nicht gleich Wasserstoff ist und wenn ferner R^3 fehlt, R^4 mindestens 5 Kohlenstoffatome enthalten muß,

R^5 ein Wasserstoffatom oder eine Gruppe der Formel $-COOA^4$ bedeutet,

R^6 Wasserstoff oder C_1 - C_4 Alkyl bedeutet und

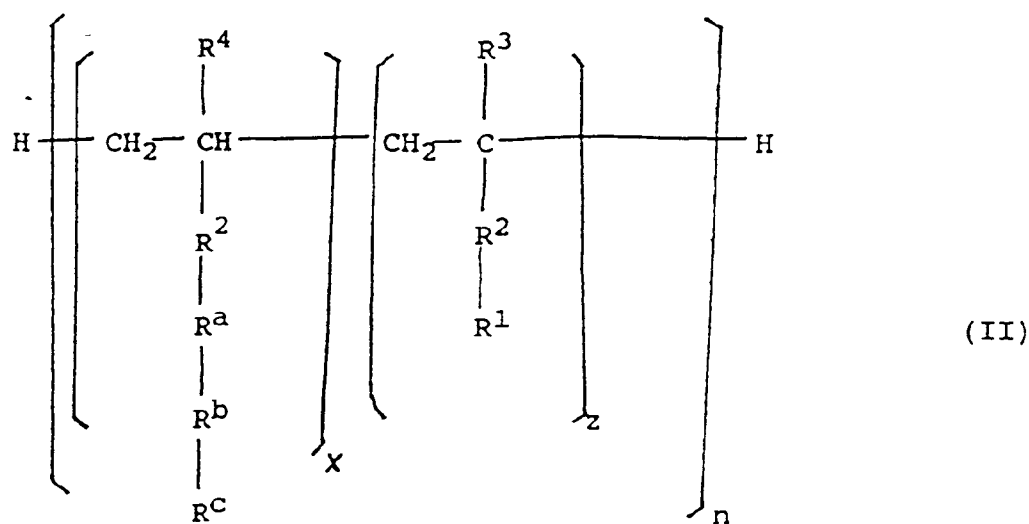
A^1 , A^2 , A^3 und A^4 unabhängig voneinander aus Wasserstoff, Alkalimetallen, Erdalkalimetallen, Ammonium- und Aminbasen sowie C_1 - C_4 oder $(C_2H_4O)_tH$ mit t ist gleich 1-50 ausgewählt sind und wobei die Monomereinheiten in Zufallsreihenfolge vorliegen können,

Q^1 ein multifunktionelles Monomer, das die Verzweigung des Polymeren gewährleistet, wobei die Monomeren des Polymeren mit Q^1 in jeder beliebigen Richtung und in jeder beliebigen Reihenfolge unter eventueller Bildung eines verzweigten Polymeren verbunden sein können, vorzugsweise Trimethylpropantri-acrylat (TMPTA), Methylenbisacrylamid oder Divinylglykol,

n mindestens 1, z und $v = 1$ und $(x + y + p + q + r)/z$ 4/1 bis 1000/1, vorzugsweise 6/1 bis 250/1, wobei die Monomereinheiten in Zufallsreihenfolge vorliegen können, vorzugsweise entweder p und $q = 0$ oder $r = 0$, insbesondere p , q , y und $r = 0$,

R^7 und R^8 $-CH_3$ oder $-H$ und

R^9 und R^{10} Substituentengruppen, beispielsweise Amino-, Amin-, Amid-, Sulfonat-, Sulfat-, Phosphonat-, Phosphat-, Hydroxy-, Carboxyl- und Oxidgruppen, vorzugsweise ausgewählt aus $-SO_3Na$, $-CO-O-C_2H_4-OSO_3Na$, $-CO-O-NH-C(CH_3)_2-SO_3Na$, $-CO-NH_2$, $-O-CO-CH_3$, $-OH$;



worin x , z und n die oben angegebene Bedeutung besitzen,

R^3 und R^4 für Wasserstoff oder C_1 - C_4 Alkyl steht,

R^2 $-CO-O-$, $-O-$, $-O-CO-$, $-CH_2-$, $-CO-NH-$ bedeutet oder fehlt,

R^1 für $-C_3H_6-N^+-(CH_3)_3(Cl^-)$,

$-C_2H_4-OSO_3^-(Na^+)$, $-SO_3^-(Na^+)$,

$-C_2H_4 N^+(CH_3)_3 Cl^-$, $-C_2H_4 N^+(C_2H_5)_3 Cl^-$,

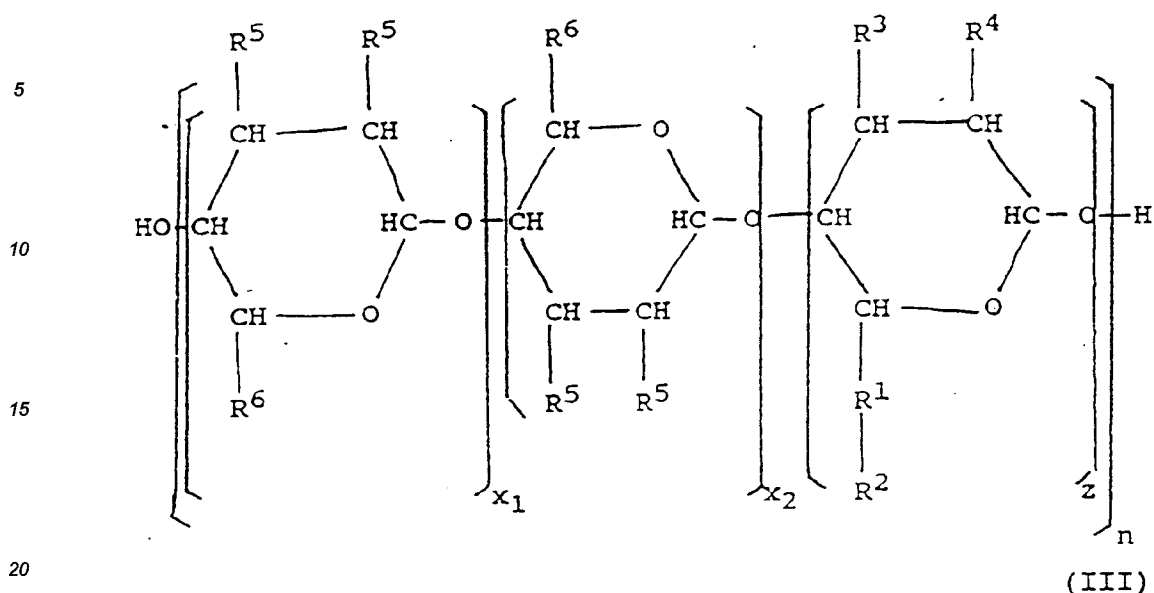
$-CH_2 N^+(CH_3)_3 Cl^-$, $-CH_2 N^+(C_2H_5)_3 Cl^-$

oder Benzyl- $SO_3^-Na^+$ darstellt,

R^a CH_2 , C_2H_4 , C_3H_6 bedeutet oder fehlt,

R^b 1 bis 50 unabhängig ausgewählte Alkylenoxidgruppen, vorzugsweise Ethylenoxidgruppen darstellt oder fehlt,

R^c $-OH$ oder $-H$ entspricht und wobei, wenn R^2 , R^a und R^b fehlen, R^c nicht für $-H$ steht;



worin bedeuten:

$x = x_1 + x_2$,

x , z und n die oben angegebene Bedeutung,

R^1 für $-\text{CH}_2\text{O}-$ oder $-\text{O}-$,

R^2 $-\text{CH}_2\text{COO}^-\text{Na}^+$, $-\text{C}_3\text{H}_6\text{N}^+(\text{CH}_3)_3\text{C1}^-$ oder $\text{C}_3\text{H}_6\text{ON}^+(\text{CH}_3)_3\text{C1}^-$,

R^3 und R_4 $-\text{OH}$, CH_2OH , $-\text{O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$, $-\text{CH}_2\text{-O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$ oder $-\text{OCH}_2\text{COO}^-\text{Na}^+$, $-\text{O-C}_3\text{H}_6\text{ON}^+(\text{CH}_3)_3\text{C1}^-$

oder $-\text{O-C}_3\text{H}_6\text{N}^+(\text{CH}_3)_3\text{C1}^-$

R^5 $-\text{OH}$, $-\text{NH-CO-CH}_3$ oder $-\text{O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$,

R^6 $-\text{OH}$, CH_2OH , $-\text{CH}_2\text{-OCH}_3$, $-\text{O}(\text{C}_3\text{H}_6\text{O})_p\text{-H}$ oder $-\text{CH}_2\text{O}-(\text{C}_3\text{H}_6\text{O})_p\text{-H}$ und

p 1-10.

5. Zusammensetzung nach Anspruch 1 oder 2 mit einem Volumen der lamellaren Phase von weniger als 0,55.
6. Zusammensetzung nach Anspruch 1 oder 2, die bei 21-tägiger Lagerung vom Zeitpunkt ihrer Herstellung ab bei 25°C zu weniger als 10 Vol.-% Phasentrennung führt, wie es durch Auftreten von zwei oder mehr getrennten Phasen augenscheinlich wird.
7. Zusammensetzung nach Anspruch 1 oder 2 mit einer Viskosität von weniger als 2500 mPa·s bei 21s⁻¹.
8. Zusammensetzung nach Anspruch 1 oder 2, umfassend 1 bis 70 Gew.-% reinigungsmittelaktiver Materialien, 1 bis 60 Gew.-% aussalzender Elektrolyten und 0,01 bis 5 Gew.-% entflocculender Polymerer.

Revendications

1. Composition détergente liquide qui comprend une dispersion de gouttelettes lamellaires de matériaux détergents actifs au sein d'une phase aqueuse continue, ladite composition comprenant également un polymère défloculant et des particules en suspension d'une matière solide, dans laquelle au moins l'une des conditions suivantes doit être satisfaite :

(1) au moins 50% des gouttelettes lamellaires ont un diamètre inférieur à 0,45 µm ;

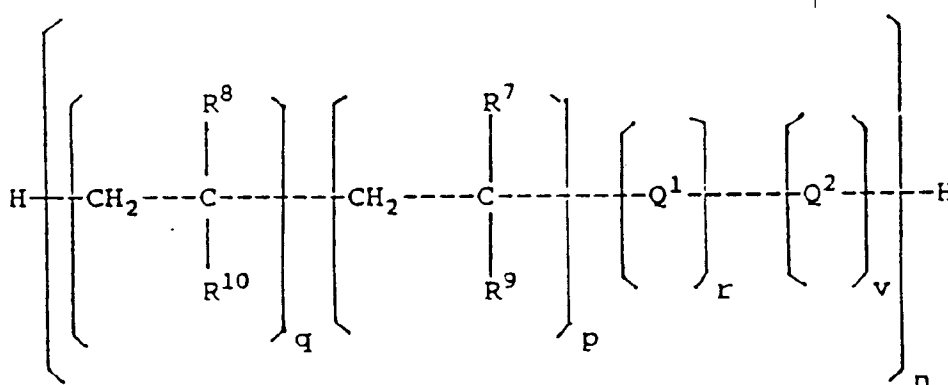
(2) la composition détergente possède un indice de réfraction pour la lumière d'une longueur d'onde de 589 nm qui est supérieur d'au moins 0,01 à l'indice de réfraction de sa phase aqueuse continue correspondante.

2. Composition détergente liquide qui comprend une dispersion de gouttelettes lamellaires de détergents

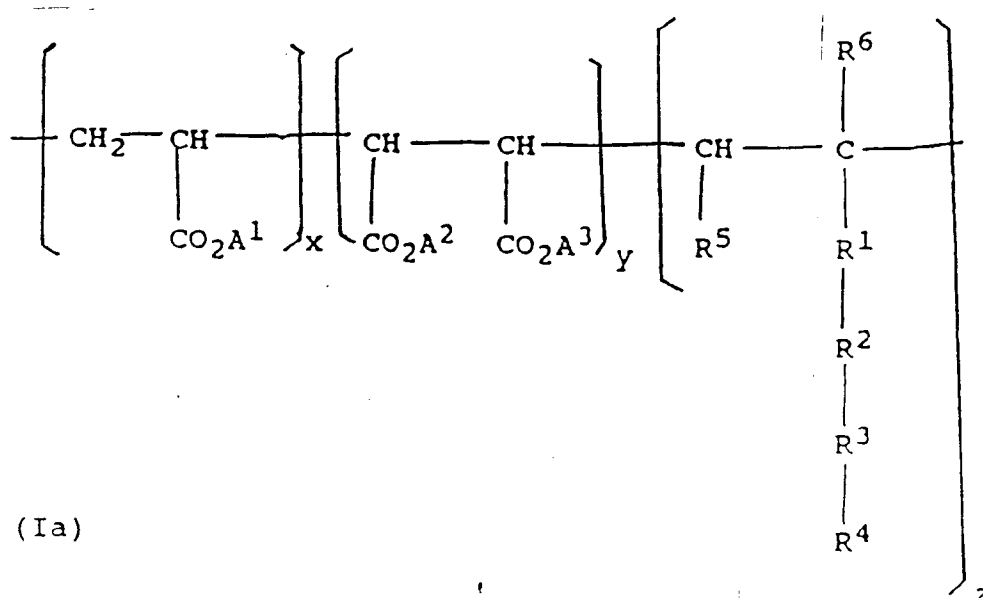
actifs au sein d'une phase aqueuse continue, ladite composition comprenant un polymère défloculant et ladite composition étant obtenable par un procédé consistant à mélanger les détergents actifs dans l'eau (facultativement en présence d'autres ingrédients de la composition) à un taux de cisaillement de plus

3. Composition selon la revendication 2, qui comprend des particules de matières solides en suspension.
4. Composition selon la revendication 1 ou 2, dans laquelle le polymère défloculant est de formule I, II ou III comme spécifié ci-après :

Formule I :



dans laquelle Q² est un ensemble moléculaire de formule Ia:



dans laquelle :

R¹ représente -CO-O-, -O-, -O-CO-, -CH₂-, -CO-NH- ou est absent ;

R² représente de 1 à 50 groupes alkylène-oxy indépendamment choisis, de préférence des groupes oxyde d'éthylène ou oxyde de propylène, ou est absent, à la condition que lorsque R³ est absent et R⁴ représente

un atome d'hydrogène ou ne contient pas plus de 4 atomes de carbone, alors R^2 doit contenir un groupe alkylèneoxy, de préférence 5 groupes alkylèneoxy d'au moins 3 atomes de carbone;

R^3 représente une liaison phénylène ou est absent ;

R^4 représente un atome d'hydrogène ou un radical alkyle en C_{1-24} ou alcényle en C_{2-24} , aux conditions que :

a)

quand R^1 représente $-O-CO-$, R^2 et R^3 doivent être absents et R^4 doit contenir au moins 5 atomes de carbone ;

b)

quand R^2 est absent, R^4 n'est pas un atome d'hydrogène et si R^3 est également absent, alors R^4 doit contenir au moins 5 atomes de carbone ;

R^5 représente un atome d'hydrogène ou un groupe de formule $-COOA^4$;

R^6 représente un atome d'hydrogène ou un radical alkyle en C_{1-4} ; et

A^1, A^2, A^3 et A^4 sont indépendamment choisis parmi les atomes d'hydrogène, les métaux alcalins, les métaux alcalino-terreux, l'ammonium et les bases amine et C_{1-4} ,

ou $(C_2H_4O)_tH$ dans lequel t est de 1 à 50, et les motifs monomères peuvent être en tout ordre aléatoire.

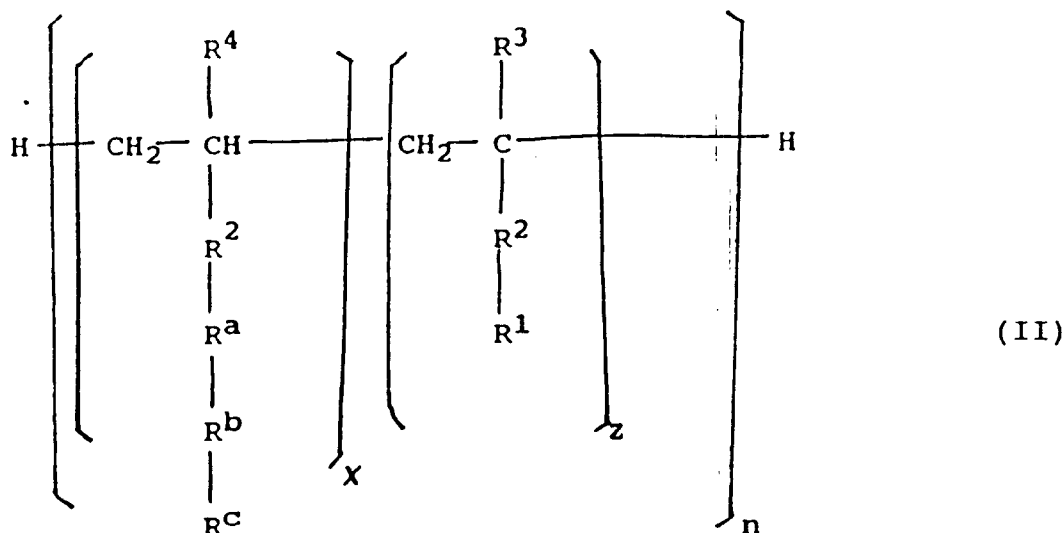
Q^1 est un monomère multifonctionnel permettant la ramification du polymère, les monomères du polymère pouvant être reliés à Q^1 suivant une direction quelconque et en un ordre quelconque, pour donner éventuellement un polymère ramifié. De préférence, Q^1 est le triacrylate de triméthylpropane (TMPTA), le méthylène-bis-acrylamide ou le divinyl-glycol,

n est au moins 1 ; z et v sont 1 ; et le rapport $(x + y + p + q + r) : z$ est de 4:1 à 1000:1, de préférence de 6:1 à 250:1 ; les motifs monomères peuvent être dans un ordre fortuit, et de préférence p et q sont zéro, ou bien r est zéro ; on préfère particulièrement que p, q, y et r soient zéro.

R^7 et R^8 représentent $-CH_3$ ou $-H$;

R^9 et R^{10} représentent des groupes substituants tels que des groupes amino, amine, amide, sulfonate, sulfate, phosphonate, phosphate, hydroxy, carboxyle et oxyde, ils sont de préférence choisis parmi :

$-SO_3Na$, $-CO-O-C_2H_4-OSO_3Na$, $-CO-O-NH-C(CH_3)_2-SO_3Na$, $-CO-NH_2$, $-O-CO-CH_3$, ou $-OH$;



dans laquelle : x, z et n sont comme ci-dessus,

R^3 et R^4 représentent H ou alkyle en C_{1-4} ;

R^2 représente $-CO-O-$, $-O-$, $-O-CO-$, $-CH_2-$, $-CO-NH$, ou est absent ;

R^1 représente : $-C_3H_6-N^+(CH_3)_3(Cl^-)$,

$-C_2H_4-OSO_3^-(Na^+)$, $-SO_3^-(Na^+)$,

$-C_2H_4-N^+(CH_3)_3Cl^-$, $-C_2H_4-N^+(C_2H_6)_3Cl^-$,

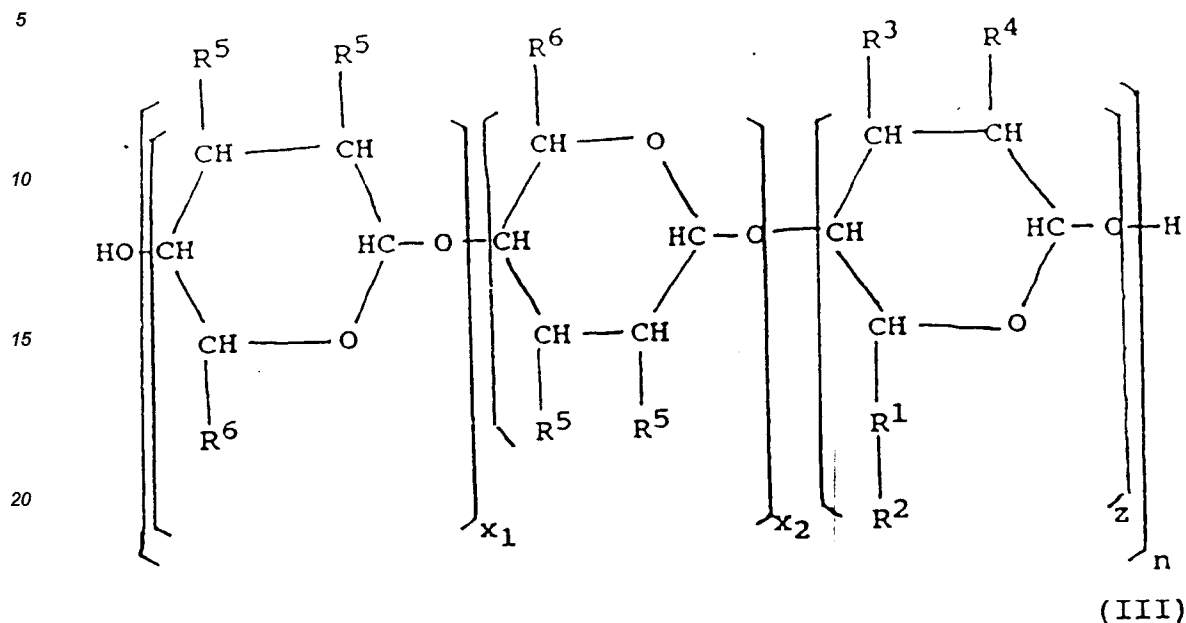
$-CH_2-N^+(CH_3)_3Cl^-$, $-CH_2-N^+(C_2H_6)_3Cl^-$ ou benzyl $-SO_3^-Na^+$;

R^a représente CH_2 , C_2H_4 , C_3H_6 ou est absent ;

R^b représente de 1 à 50 groupes indépendamment choisis d'oxyde d'alkylène, de préférence d'oxyde d'éthylène ou est absent ;

R^c représente -OH ou -H ;

et quand R², R^a et R^b sont absents, R^c n'est pas -H.



dans laquelle :

$$x = x_1 + x_2$$

x, z et n sont comme ci-dessus

R¹ représente -CH₂O- ou -O- ;

R² représente -CH₂COO⁻Na⁺, -C₃H₆N⁺(CH₃)₃Cl⁻ ou -C₃H₆ON⁺(CH₃)₃Cl⁻

R³ et R⁴ représentent :-OH, CH₂OH, -O(C₃H₆O)_p-H, -CH₂-O(C₃H₆O)_p-H ou -OCH₂COO⁻Na⁺ -O-C₃H₆ON⁺(CH₃)₃Cl⁻ ou -O-C₃H₆N⁺(CH₃)₃Cl⁻

R⁵ représente -OH, -NH-CO-CH₃ ou -O(C₃H₆O)_p-H

R⁶ représente -OH, -CH₂OH, -CH₂-OCH₃, -O(C₃H₆O)_p-H ou -CH₂-O-(C₃H₆O)_p-H

p est de 1 à 10.

5. Composition selon la revendication 1 ou 2, dont le volume de phase lamellaire est inférieur à 0,55.

6. Composition selon la revendication 1 ou 2, donnant moins de 10% en volume de séparation de phases comme on peut le constater par l'aspect de deux ou plusieurs phases séparées, quand on les emmagasine à 25°C pendant 21 jours à partir du moment de leur préparation.

7. Composition selon la revendication 1 ou 2, dont la viscosité à 21 s⁻¹ est inférieure à 2500 mPa.s.

8. Composition selon la revendication 1 ou 2, comprenant de 1 à 70% en poids de détergents actifs, de 1 à 60% en poids d'électrolytes de relargage et de 0,01 à 5% en poids de polymères défloculants.