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(54) **DETERGENT COMPOSITION**

WASCHMITTELZUSAMMENSETZUNG

COMPOSITION DETERGENTE

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WO-A-94/01524

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Description

Technical Field

[0001] The present invention relates to compositions which comprise polymer material and to a method of preparing such compositions.

Background & Prior Art

[0002] Detergent compositions may be aqueous or non-aqueous, both either in solid or in liquid form. Non-aqueous liquid detergent compositions are known in the art and have been described in quite a number of patent publications, e.g. in US-A-4,316,812, US-A-4,874,537 and EP-A-0,484,095. Generally, the free water content of non-aqueous liquid detergent compositions is less than 5% by weight, preferably less than 3% by weight, more preferably less than 2% by weight, most preferably substantially nil. Non-aqueous liquid detergent compositions provide a way of concentrating liquid detergents without giving in on washing performance.

[0003] Non-aqueous liquid detergent compositions normally comprise a non-aqueous liquid phase having incorporated therein as dispersion, solution or combination thereof, the usual detergent components and adjuncts depending on the purpose of use, primarily surfactants and builders.

[0004] The non-aqueous liquid phase often comprises a nonionic surfactant as major component, which apart from acting as carrier liquid for the other detergent components, usually and preferably also has detergent-active properties, thereby acting wholly or in part as the surfactant ingredient.

[0005] One weakness of non-aqueous liquid detergent compositions is that on dilution with water, they may tend to suffer from gelling and the ingredients tend to stick together. As a consequence thereof, the product may disperse only slowly in water and this may have a negative influence on the cleaning performance of the product, e.g. the product will not uniformly and timely dissolve in the washing liquor. This is for example true with liquid phases comprising a nonionic surfactant, such as mixtures of C13-C15 alcohols with an average of 3 ethoxy groups and C13-C15 alcohols with an average of 7 ethoxy groups and liquid phases as described in GB-A-1,462,134, WO-A-91/12313, WO-A-91/14765 and EP-A-0,510,762 and more in particular for liquids comprising a dispersed solid particulate phase.

[0006] Such problems have for example been described in GB-A-2,169,613 (Colgate) wherein alkylene glycol mono alkyl ethers are disclosed as anti-gelling agent. GB-A-2,168,995 (Colgate) describes acid-terminated nonionics and GB-A-2,177,716 (Colgate) dicarboxylic acids as gel-inhibition agents. Other examples of anti-gelling compounds, such as lower aliphatic alcohols, glycols and polyethyleneglycol, are described in EP-A-413,616 (Colgate). Our co-pending PCT Applica-

tion No. EP/94/01289 (Unilever) describes the use of an ester material to overcome gelling problems.

[0007] Indeed, it has been found that these solvent materials may be beneficial in at least partially overcoming the gelling problem of non-aqueous liquid detergent compositions. However, we have found that these materials are only effective to overcome gelling caused at relatively high water levels, whereas they are less effective or even ineffective in overcoming relatively low water level gelling problems, e.g. at water levels in which 5 to 50% by weight of water is added to the product.

[0008] Therefore, there still exists a gelling problem of non-aqueous liquid detergent compositions at relatively low water levels, say water levels of 5 to 50% by weight of the composition. Additionally, gelling problems of non-aqueous liquid detergent compositions at relatively high water levels, say water levels of 70 to 250% by weight of the composition, can still occur.

[0009] Gelling problem of non-aqueous liquids are not only noticeable for non-aqueous liquid containing a non-aqueous phase, but also, and in particular, when solid dispersed particles are present.

[0010] Further, dispersion problems of non-aqueous liquid detergent compositions may occur upon dilution with water. Low dispersion may for example lead to high concentrations of product on the cloth that may have negative effects on the cloth's quality.

[0011] A further problem related to the use of non-aqueous liquid compositions is dissolution. Slow dissolution of the concentrated composition may result in liberation of the components to the wash liquor only at a late stage during the washing process, at which point they may be less effective.

[0012] Another problem that is in particular important in the industrial area is the dispensing problem of non-aqueous liquids, i.e. the blocking of pipes and valves of the dosing system for non-aqueous liquid detergent compositions. This problem has e.g. been described in EP-A-480,490 (Unilever).

[0013] EP-A-413,616 (Colgate) discloses non-aqueous liquid detergent compositions which are storage-stable, easily pourable and dispersible in cold, warm or hot water. These compositions comprise certain polymers which are derived from α,β -monoethylenically unsaturated carboxy-containing monomers which also contain at least one other chalcogen-containing group substituted with at least one group of at least two carbon atoms.

[0014] It is an object of the present invention to improve at least one of the problems related to gelling, dispersion, dissolution and/or dispensing of non-aqueous liquids.

[0015] Surprisingly, we have found that the above mentioned problems of gelling, dispersion, dissolution and/or dispensing may at least partially be overcome by including a particular polymeric material.

[0016] Non-aqueous liquids comprising polymeric material have for example been described in EP-A-

510,762 (Unilever) that discloses non-aqueous liquids comprising a polymer material comprising at least one group capable of association with the solid phase and at least one group capable of extending from the solid phase. The polymer is amongst other incorporated to lower the viscosity and the clear layer separation.

[0017] WO 94/01524 (Unilever) discloses non-aqueous liquids comprising a polymeric material with a group that exhibits self association to reduce viscosity and the clear layer formation of the liquids.

[0018] WO 94/12611 (Unilever) discloses non-aqueous liquids comprising dry particulate solubilised biopolymer material to improve the dispersion of the liquid. Preferred biopolymers are polysaccharides and polypeptides.

[0019] EP-A-359,491 (Unilever) and EP-A-359,492 (Unilever) disclose non-aqueous liquids comprising respectively low and high molecular weight vinyl pyrrolidone polymers.

[0020] EP-A-28,849 (Unilever) discloses non-aqueous liquids comprising a stabilising polymer that is at least partially hydrolysed copolymer of maleic anhydride with ethylene or vinylmethylether.

[0021] GB-A-2,228,944 (Unilever) discloses non-aqueous liquids comprising polymeric materials that may comprise acryl amide monomers.

Statement of the invention

[0022] The present invention provides a non-aqueous liquid detergent composition according to claim 1. Preferably, the non-aqueous liquids according to the invention comprise solid dispersed particles which are more preferably selected from bleaches, builders, abrasives, dispersants, enzymes, solid surfactants, fluorescers, enzymes, bleach precursors, bleach catalysts, colorants, pigments, opacifying agents and mixtures thereof.

[0023] The invention further provides a method of preparing a non-aqueous liquid detergent composition comprising a non-aqueous phase and a polymeric hydrotrope, according to claim 4.

Description of the Invention

[0024] It has been found that non-aqueous liquids comprising the polymeric hydrotrope show reduced gelling characteristics upon dilution, improved dispersion, dissolution and/or dispensing.

[0025] It has further been found that the use of the polymeric hydrotrope in non-aqueous liquids according to the invention may have certain advantages compared with use of conventional hydrotropes, including low foaming properties, good surfactant solubilising characteristics, chemical stability, ease of handling, cost effective, substantially free of nitrosamines commonly associated with compositions using nitrogen-based hydrotropes or solubilisers, such as amine oxides.

[0026] Further, use of the polymer hydrotrope has

been found to lead to reduced gelling, improved dispersion, dissolution and/or dispensing of compositions comprising high surfactant levels.

[0027] Mixing the polymeric hydrotrope in a composition has been found to be advantageous as to reduced gelling characteristics upon dilution, improved dispersion, dissolution and/or dispensing when applying the composition to a washing machine.

[0028] Mixing a surfactant-composition with a polymeric hydrotrope leads to reduced gelling characteristics upon dilution, improved dispersion, dissolution and/or dispensing when mixing the composition with water.

Polymeric Hydrotrope

[0029] The polymeric hydrotrope may be a copolymer, in block and/or random form. The polymer comprises hydrophilic and hydrophobic monomers.

[0030] The Hydrophilic monomer is acrylic acid or methacrylic acid.

[0031] Preferably, the molar ratio between the hydrophilic and the hydrophobic monomers in the polymer is from 5:1 to 1:5, preferably 3:1 to 1:3, more preferably 2:1 to 1:2.

[0032] The hydrophobic monomer is styrene.

[0033] One preferred polymer comprises acrylic acid and styrene, possibly with minor amounts of dodecylmercaptan (DDM). Acrylic acid is preferably present in an amount in the range 50-80% by wt, the styrene is preferably present in an amount in the range 20-50 % by weight and DDM is preferably present in an amount up to about 1% by weight, eg. 0.1-1% by weight.

[0034] A particularly preferred polymer comprises 50.81 wt% acrylic acid, 48.93 wt% styrene, 0.26 wt% DDM, with a molecular weight of about 1,000 and substantially no cross-linking. This polymer is found to be very versatile and to have a wide range of uses and applications.

[0035] Another preferred polymer comprises 54 wt% methacrylic acid and 46% styrene, with a molecular weight of about 100,000 and substantially no cross-linking. Such a polymer is found to be hypochlorite-stable and to be an effective thickener in hypochlorite bleach.

[0036] For most applications the polymer is preferably substantially non cross-linked; although a degree of cross-linking can be tolerated.

[0037] The polymer typically has a molecular weight of between about 1,000 and 1,000,000. For many purposes polymers with molecular weights at the bottom end of the range are preferred, as these generally make better hydrotropes. For example, a polymer with a molecular weight of about 1000 is found to be very versatile and useful in a wide range of applications. Polymers with higher molecular weights, eg. about 100,000, also function well as hydrotropes and may additionally perform a thickening function which may be desirable in certain applications, eg. production of thickened compositions.

[0038] Preferably, the molecular weight of polymers

according to the invention is at least 500, more preferably at least 750 and preferably at most 10,000, more preferably at most 5,000, most preferably at most 3,000, e.g. 1,000.

[0039] Suitable polymers can be readily made in conventional manner, eg. by emulsion or solution polymerisation, and are preferably in the form of low viscosity non-aqueous solutions or powders.

[0040] The polymer is present in an appropriate amount to achieve desired results, such as particular desired composition properties, and suitable amounts can be readily determined by experiment. Typically amounts in the range 0.1 to 15% by wt as active material are suitable, preferably 0.5 to 10% by wt.

Electrolyte material

[0041] Electrolyte material is incorporated in compositions according to the invention.

[0042] For the purpose of this invention electrolyte material is defined as material that dissociates at least for 10% by weight of the total amount of electrolyte upon dilution of 1g in 100ml of water at 25°C. The electrolyte material may include builder, bleach or other materials.

[0043] Preferably, the electrolyte material according to the invention is ionisable and water soluble. More preferably, the electrolyte material is an alkaline material, i.e. the salt form of the electrolyte has a pH in water at 25°C and 1 atm of higher than 7, preferably 7.5 or higher, more preferably 8.0 or higher.

[0044] In particular it is preferred that the electrolyte material is selected from salts (e.g. alkali metal) organic and mineral acids. Examples of such electrolyte material are phosphates (e.g. STP), silicates (eg. metasilicates, disilicates), hypochlorites, carbonates (e.g. bicarbonates and percarbonates), hydroxides, halides (e.g. chloride), sulphates (e.g. sulphate), sequestrants (e.g. ethylenediamine-tetracetic acid and sodium nitrilotriacetate), citrates and borates (e.g. perborates). Equally, the corresponding acids can be used, as these generally turn into the effective salt form in the non-aqueous liquid product itself or immediately upon dilution in the wash solution.

[0045] Electrolyte is typically present in an amount of at least 0.5%, preferably 1.0%, more preferably 2% and preferably at most 60%, more preferably at most 50% by weight of the composition.

Non-Aqueous Liquid Phase

[0046] Composition of the present invention may comprise surfactant material, non-surfactant material, solvent material and other liquid material such as deflocculant material.

[0047] Compositions of the invention may comprise from 10 to 90% by weight of a non-aqueous liquid phase. Preferably the level of the liquid phase is at most 75%, or at most 70% and preferably, at least 20%, more pref-

erably at least 30%.

[0048] Non-aqueous liquids to which the hydrotrope may be added contain a non-aqueous phase comprising a single surfactant or a mixture of surfactants selected from a wide range, including the following.

Surfactants

[0049] Detergent compositions according to the invention may comprise anionic, nonionic, cationic, amphoteric and/or zwitterionic surfactant material, preferably in levels of from 10 to 90% by weight of the composition.

[0050] Typical blends of surfactants include a nonionic and/or non-alkoxylated anionic and/or alkoxylated anionic surfactant. Cationic, zwitterionic and amphoteric surfactants may also be present in minor amounts as desired. These and other surfactants are 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, Munchen & Wien.

[0051] Compositions comprise a liquid nonionic surfactant. Nonionic detergent surfactants are well-known in the art. They normally consist of a water-solubilising polyalkoxylene (preferably from 3-10 ethoxy and/or propoxy groups) or a mono- or di-alkanolamide group in chemical combination with an organic hydrophobic group derived from, for example, fatty alcohols with from 9 to 15 carbon atoms (optionally branched, e.g. methyl branched), alkylphenols (preferably from 12 to 20 carbon atoms) in which the alkyl group contains from about 6 to about 12 carbon atoms, dialkylphenols in which each alkyl group contains from 6 to 2 carbon atoms, primary, secondary or tertiary aliphatic alcohols (or alkyl-capped derivatives thereof), monocarboxylic acids having from 10 to about 24 carbon atoms in the alkyl group and polyoxypropylenes.

[0052] Fatty acid mono- and dialkanolamides in which the alkyl group of the fatty acid radical contains from 10 to about 20 carbon atoms and the alkyloyl group having from 1 to 3 carbon atoms are also common. In any of the mono- and dialkanolamide derivatives, optionally, there may be a polyoxyalkylene moiety joining the latter groups and the hydrophobic part of the molecule.

[0053] In all polyalkoxylene containing surfactants, the polyalkoxylene moiety usually consists of an average of from 2 to 20 groups of ethylene oxide, propylene oxide groups or mixtures thereof. The latter class includes those described in European Patent Specification EP-A-0,225,654, especially for use as all or part of the liquid phase.

[0054] Especially preferred are those ethoxylated nonionics which are condensation products of fatty alco-

hols with from 9 to 15 carbon atoms condensed with 3 to 7 moles of ethylene oxide. Examples of those are the condensation products of C₁₁₋₁₃ alcohols with 3 or 7 moles of ethylene oxide. These may be used as the sole nonionic surfactant or in combination with those described in EP-A-0,225,654. Suitable examples of such nonionics are Vista 1012-6.2, which is a C₁₀₋₁₂ alcohol ethoxylated with on average 6.2 ethoxy groups, and Dobanol 25-3, which is a C₁₀₋₁₅ alcohol ethoxylated with on average 3 ethoxy groups.

[0055] Another class of suitable nonionics include the alkyl saccharides (polyglycosides/oligosaccharides) and, in particular those described in the following patent specifications, US-A-3,640,998; US-A-3,346,558; US-A-4,223,129; EP-A-0,092,355; EP-A-0,099,183; EP-A-0,070,074; EP-A-0,070,075; EP-A-0,070,075; EP-A-0,070,076; EP-A-0,070,077; EP-A-0,075,994; EP-A-0,075,995 and EP-A-0,075,996.

[0056] Mixtures of different nonionic detergent surfactants may also be used. Mixtures of nonionic detergent surfactants with other detergent surfactants such as anionic, cationic or ampholytic surfactants and soaps may also be used.

[0057] The level of nonionic surfactant is from 10 to 90% by weight of the composition, more preferably from 20 to 70% by weight of the composition and, most preferably, from 35 to 50% by weight of the composition.

Solvent Material

[0058] Solvent material may be included in compositions according to the present invention and in particular materials as disclosed in our co-pending PCT application EP/94/01289, i.e. an ester of a C₁₋₆ carboxylic acid and/or polycarboxylic acid and an alcohol selected from dihydric alcohols, ether alcohols and sterically hindered alcohols. Preferably, the ester is present in amounts of from 0.01 to 50% by weight of the composition. A suitable ester material is propyleneglycoldiacetate. Other examples are disclosed in GB-A-2,169,613, GB-A-2,177,716 and EP-A-413,616 (Colgate).

Other Liquid Material

[0059] Other liquid material which may be present in the liquid phase include liquid bleach precursors such as for example glyceroltriacetate, solvent material, for example ethanol and dodecanol, and deflocculant material such as described in EP-A-0,266,199.

[0060] The level of liquid precursors is preferably 0 to 20% by weight, more preferably 0 to 10% by weight. The level of solvents, other than nonionic surfactants, is preferably from 0 to 20% by weight, more preferably 0 to 10%.

[0061] In principle, any material may be used as a deflocculant provided it fulfils the deflocculation test described in European Patent Specification EP-A-0,266,199 (Unilever). The capability of a substance to

act as a deflocculant will partly depend on the solids/liquid phase combination. However, especially preferred are acids.

[0062] Deflocculants are preferably selected from Bronsted acids and Lewis acids. Examples thereof are transition metals, anhydrides, organic acids, inorganic mineral acids and salts, C₈-C₂₀ fatty acids and salts, C₁-C₁₀ alkanolic acids, substituted anhydrous alkali-metal aluminosilicate, alkyl, alkenyl aryl, aralkyl and aralkenyl sulphonic or carboxylic acids and salts and halogenated derivatives thereof, zwitterionic surfactants, anionic surfactants of the formula R-L-A-Y, as defined in EP-A-0,266,199, and compounds having an acidic -P-OH group. Preferably, deflocculants in the acid form are used.

[0063] Deflocculant material, if included, may be present at levels of from 0 to 15%, preferably at least 0.01 and most preferably at least 1% by weight. For most purposes, the amount of deflocculant material will be from 2 to 12 % by weight, preferably 4 to 10% by weight based on the final composition.

Solid Particles

[0064] Composition according to the invention may be isotropic (i.e. may not contain solid particles) or they may comprise solid (dispersed) particles in which case the solid phases may include one or more components selected from abrasives, bleaches, bleach catalysts, colorants, bleach precursors, builders, dispersants, enzymes, fluorescers, opacifying agents, pigments, solid surfactants and mixtures thereof.

[0065] Preferably, compositions according to the invention contain from 0 to 90% by weight of a solid phase. Preferably, the level of the solid phase is at least 10%, more preferably at least 20%. Preferably, at most 80%, more preferably at most 70%, in particular at most 60%.

[0066] Usually (at least part of) the particle size of the solid phase in terms of D(3,2) will be less than 500, preferably not more than 250µm, more preferably not more than 100µm. Preferably, the particle size is at least 0.1µm. D(3,2) refers to average particle diameter, which is an average surface weighted, volume/weight mean diameter.

Bleach system

[0067] Optionally compositions of the present invention may comprise a bleach system. Suitable bleaches for inclusion in the detergent compositions of the invention include halogen, particularly chlorine bleaches, such as are provided in the form of alkali-metal hypohalites (e.g. hypochlorites), as well as peroxygen bleach. When the compositions of the invention are to be used for fabric washing, peroxygen bleaches are preferred, for example, in the form of an inorganic peroxybleach, optionally in combination with one or more bleach activators.

[0068] Typical examples of suitable peroxy bleach are alkali-metal perborates, both tetrahydrates and monohydrates, alkali metal percarbonates, persulphates, persilicates, perphosphates and peracids. Perborate and percarbonate are preferred, in particular in the sodium form.

Activators

[0069] Compositions according to the invention comprise preferably additionally to the peroxygen bleach, a bleach activator, which makes the bleach more effective at lower temperatures, i.e. in the range from ambient temperature to about 60°C. Bleach activators may be selected from bleach precursors, bleach catalysts and mixtures thereof.

[0070] Peroxygen bleach such as sodium perborate, both the monohydrate and the tetrahydrate, acts to release active oxygen in solution, and the precursor which is usually an organic compound having one or more reactive acyl residues which causes the formation of peroxy acids; the latter providing for more effective bleaching action at lower temperatures than the peroxybleach compound alone.

[0071] Precursors according to the invention include compounds with N- and O-acyl groups. Suitable classes include anhydrides, esters, imides and acylated derivatives of imidazoles and oximes, and examples of useful materials within these classes are disclosed in GB-A-1,586,789, GB-A-0,836,988, GB-A-0,864,798, GB-A-1,147,871, GB-A-2,143,231 and GB-A-0,855,735 and GB-A-1,246,338. Specific O-acetylated precursor compounds include 2,3,3-tri-methyl hexanoyl oxybenzene sulfonates, benzyl oxybenzene sulfonates and penta acetyl glucose. Particularly preferred precursor compounds are the N-,N,N1N1 tetra acetylated compounds of which tetra acetyl methylene diamine (TAMD), tetraacetyl ethylene diamine (TAED) and tetraacetyl hexylene diamine (TAHD) are preferred examples. The ratio of the peroxybleach to the precursor is from 20:1 to about 1:1. The preferred level of the peroxybleach compound in the composition is from 0 to 30% by weight. The preferred level of precursor is from 0 to 20% by weight of the composition.

[0072] Inclusion of bleach catalysts and, in particular, transition metal catalysts may also be advantageous. Such catalysts, optionally together with stabilisers, as hereinafter defined, can be used to activate peroxide compounds to make them more suitable for use for bleaching at lower temperatures, e.g. from 20 to 60°C. As stated above, such catalysts may be incorporated in the form of a particulate product according to the invention. Such catalysts, which only need be present in such detergent composition in small amounts such as from 0.005 to 5% by weight of the composition.

[0073] Bleach catalysts may include those based on metal ion delivered by simple salts such as Cu(II) sulphate. Preferred bleach catalysts are based on transi-

tion metal coordination complexes as has been described and defined in EP-A-0,458,397, EP-A-0,458,398, EP-A-0,522,817, EP-A-0,544,440, EP-A-0,544,490, EP-A-0,549,272, EP-A-0,549,271, EP-A-0,544,491, US-A-5,194,416, WO 93/25562, WO 94/00439, and WO 94/05422.

[0074] Other compounds that may be used in compositions according to the present invention are oxygen transfer agents as described and defined in EP-A-446,981, EP-A-453,003 and EP-A-446,982.

[0075] It may also be desirable to include in the compositions a stabiliser for the bleach or bleach system, for example hydroxyethylidene-1,1-diphosphonic acid, ethylene diamine tetramethylene phosphonate and diethylene triamine pentamethylene phosphonate or other appropriate organic phosphonates or salts thereof, such as the Dequest® range of materials.

Builders

[0076] The detergency builders are those materials which counteract the effects of calcium, or other ion, water hardness, either by precipitation or by an ion sequestering effect. They comprise both inorganic and organic builders. They may also be sub-divided into the phosphorus-containing and non-phosphorus types, the latter being preferred when environmental considerations are important.

[0077] In general, the inorganic builders comprise the various phosphate-, carbonate-, silicate-, borate- and aluminosilicates-type materials, particularly the alkali-metal salt forms. Mixtures of these may also be used.

[0078] Examples of phosphorus-containing builders are the water-soluble salts, especially alkali metal pyrophosphates, orthophosphates, polyphosphates and phosphonates. Examples of non-phosphorus-containing inorganic builders are water-soluble alkali metal carbonates, bicarbonates, borates, silicates, metasilicates, and crystalline and amorphous aluminosilicates.

[0079] Examples of organic builders include the alkali metal, ammonium and substituted ammonium, citrates, succinates, malonates, fatty acid sulphonates, carboxymethoxy succinates, ammonium polyacetates, carboxylates, polycarboxylates, aminopolycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates. Specific examples include salts of ethylenediamine-tetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, melitic acid, benzene polycarboxylic acids and citric acid.

[0080] Other suitable organic builders include the higher molecular weight polymers and co-polymers known to have builder properties, for example appropriate polyacrylic acid, polymaleic acid and polyacrylic/polymaleic acid co-polymers and their salts, such as those sold by BASF under the Sokalan^R Trade Mark. Polyacrylates or their derivatives may also be useful for their anti-ashing properties.

[0081] Preferably, the level of builder materials is from

5 to 50% by weight of the composition, more preferably 10 to 40%, most preferably 15 to 35%.

Dispersants

[0082] The physical stability of non-aqueous liquid detergent compositions can be improved and/or setting problems can be minimised, if dispersants are used.

[0083] For the purpose of the present invention, a dispersant material is a material, of which the main purpose is to stabilise the composition. Preferably, the dispersant is selected from hydrophobically modified dispersant materials, metal oxides as defined in EP-A-515,435 (preferably having a bulk density of 200 to 1000 g/l), finely divided clay material, aluminium tri-stearate and mixtures thereof.

[0084] A preferred material is hydrophobically modified (HM) silica containing dispersants are used as described and defined in EP-A-515,418.

Other Optional Ingredients

[0085] Other ingredients comprise those remaining ingredients which may be used in detergent compositions, such as fabric conditioning agents, abrasive material, enzymes, enzyme stabilising agents (if necessary), fluorescers, perfumes (including deoperfumes), micro-biocides, soil-suspending agents (anti-redeposition agent), corrosion inhibitors, and lather depressants.

[0086] Amongst the fabric conditioning agents which may be used, either in fabric washing liquids or in rinse conditioners, are fabric softening materials such as fabric softening clays, quaternary ammonium salts, imidazolinium salts, fatty amines and cellulases.

[0087] Abrasive are particularly preferred for incorporation in hard surface cleaners (liquid abrasive cleaners).

[0088] Enzymes which can be used in compositions according to the present invention include proteolytic enzymes (protease), amylolytic enzymes (amylase), lipolytic enzymes (lipases) and cellulolytic enzymes (cellulase). The preferred level of enzyme materials is from 0.01 to 5% by weight of the composition.

[0089] The total amount of the fluorescent agent or agents used in a composition is generally from 0.02 to 2% by weight.

[0090] When it is desired to include anti-redeposition agents in the liquid detergent compositions, the amount thereof is normally from about 0.1% to about 5% by weight, preferably from about 0.2% to about 2.5% by weight of the total composition.

Product Form

[0091] Compositions according to the present invention may be formulated in a very wide range of specific forms, according to the intended use. They may be formulated as cleaners for hard surfaces (with or without

abrasive) or as agents for warewashing (cleaning of dishes, cutlery etc) either by hand or mechanical means, as well as in the form of specialised cleaning products, such as for surgical apparatus or artificial dentures.

[0092] Preferably compositions of the invention are formulated as agents for washing and/or conditioning of fabrics and then the compositions contain at least one agent which promotes the cleaning and/or conditioning of the article(s) in question, selected according to the intended application. Usually, this agent will be selected from surfactants, enzymes, bleaches, builders, buffers, microbiocides, (for fabrics) fabric softening agents and (in the case of hard surface cleaning) abrasives. Of course in many cases, more than one of these agents will be present, as well as other ingredients commonly used in the relevant product form.

[0093] For the washing of fabrics, preferably an aqueous liquor containing 0.1 to 10 %, more preferably 0.2 to 2%, of the non-aqueous liquid detergent composition of the invention is used.

Processing

[0094] During manufacture, it is preferred that all equipment and raw materials should be dry and (in the case of hydratable salts) in a low hydration state, e.g. anhydrous phosphate builder, sodium perborate monohydrate and dry calcite abrasive, where these are employed in the composition. In a preferred process for preparing non-aqueous liquids, the dry, substantially anhydrous solids are blended with the liquid phase in a dry vessel. If deflocculant materials are used, these should preferably -at least partly- be mixed with the liquid phase, prior to the addition of the solids. This blend is passed through a grinding mill or a combination of mills to achieve a D(3,2) particle size of between 0.1 to 100 microns. A preferred combination of such mills is a colloid mill followed by a horizontal ball mill. Of course particulate material already having the desired particle size need not be subjected to this procedure.

[0095] During this milling procedure, the energy input results in a temperature rise in the product and the liberation of air entrapped in or between the particles of the solid ingredients. It is therefore highly desirable to mix any heat sensitive ingredients such as perfumes, enzymes and bleach components, into the product after the milling stage and a subsequent cooling step. It may also be desirable to de-aerate the product before addition of these (usually minor) ingredients and optionally, at any other stage of the process.

[0096] The present invention further relates to a method of preparing a non-aqueous liquid detergent composition comprising a polymeric hydrotrope, wherein the polymeric hydrotrope is added to the composition in a form comprising 25% or less by weight of water, preferably less 20% or less, more preferably 15% or less, most preferably 10% or less, in particular substantially non-aqueous.

[0097] Preferably the non-aqueous phase further comprises ingredients selected from abrasives, bleaches, bleach catalysts, bleach precursors, corrosion inhibitors, dispersants, enzymes, fabric softening agents, microbiocides, soil-suspending agents, solid surfactants and mixtures thereof and preferably at a level of from 0.01 to 90% by weight of the composition.

[0098] The polymeric hydrotrope can either be added as solid particles that are freeze-dried (less than 5% by weight of water), or spray-dried (5-15% water) or, preferably, in the form of a non-aqueous solution or dispersion, e.g. in nonionic surfactant material or in a solvent material.

[0099] The following examples are intended to further illustrate and describe the invention and are not intended to limit the invention in any way.

EXAMPLES

Example 1 - gelling test

[0100] 10ml product was magnetically stirred, water was added and the viscosity visually determined.

Example 1.1 - Soda

[0101] Two compositions (A and 1) were prepared with 10g Vista 1012-62 and Dobanol 25-3 (weight ratio 55/45) mixed with 2g soda. The nonionic of composition 4 additionally contained 2% of Narlex H100 polymer™ (PPE 1269, a Acrylic Acid/Styrene/DDM polymer, ex National Starch Chemical Company), hereafter the Narlex polymer. Composition 1 showed less gelling in the gelling test than composition A.

Example 1.2 - STP

[0102] Two compositions (B and 2) were prepared with 10g Vista 1012-62 and Dobanol 25-3 (weight ratio 55/45) mixed with 2g STP. The nonionic of composition 2 additionally contained 2% of the Narlex polymer. Composition 2 showed less gelling in the gelling test than composition B.

Example 1.3 - SMS

[0103] Two compositions (C and 3) were prepared with 10g Vista 1012-62 and Dobanol 25-3 (weight ratio 55/45) mixed with 2g SMS. The nonionic of composition 3 additionally contained 2% of the Narlex polymer. Composition 3 showed less gelling in the gelling test than composition C.

Example 1.4 - Percarbonate

[0104] Two compositions (D and 4) were prepared with 10g Vista 1012-62 and Dobanol 25-3 (weight ratio 55/45) mixed with 2g percarbonate. The nonionic in

composition 4 additionally contained 2% of the Narlex polymer. Composition 4 showed less gelling in the gelling test than composition D.

5 Example 1.5 - Soda/Percarbonate

[0105] Four compositions (E and 5-7) were prepared with 10g Vista 1012-62 and Dobanol 25-3 (weight ratio 55/45) mixed with 2g soda (compositions E and 5) or 2g percarbonate (composition 6 and 7). Composition 7 additionally contained 1g soda. Compositions 5-7 all contained 2% of the Narlex polymer of Example 1.1. Composition 7 showed the lowest gelling, followed by composition 5, followed by composition 6 and composition E showed the worst gelling behaviour.

Example 1.6 - Soda/STP

[0106] Four compositions (8-11) were prepared with 10g Vista 1012-62 and Dobanol 25-3 (weight ratio 55/45) mixed with 2g soda and 2% of the Narlex polymer. Compositions 8-10 all contained 1g soda. Composition 9 further contained 2g STP. Composition 10 further contained 0.5g Sodium citrate. Composition 11 contained 2g STP. All compositions showed low gelling behaviour.

Example 1.7 - Soda & Vista 1012-5.5

[0107] Two compositions (F and 12) were prepared containing 10g Vista 1012-5.5 and 2g soda. Composition 12 additionally contained 2% of the Narlex polymer of Example 1.1 and had far lower gelling behaviour than composition F.

Example 1.8 - Soda & Dobanol 25-3

[0108] Two compositions (G and 13) were prepared containing 10g Dobanol 25-3 and 2g soda. Composition 13 additionally contained 4% of the Narlex polymer and had far lower gelling behaviour than composition F (that showed lump formation).

Example 1.9 - Polymeric hydrotrope without salts

[0109] Four compositions were prepared (H and 14-16) were prepared with Vista 1012-62 with 0, 1, 2 and 4% of the Narlex polymer, respectively. Composition 16 showed the lowest viscosity, followed by composition 15, followed by composition 14. Composition H had the highest viscosity in the gelling test.

Comparative Example 1.10 - SXS

[0110] Two compositions (I and J) were prepared with Vista 1012-62 and Dobanol 25-3 (40/60 weight ratio) and 2g soda. Composition J additionally contained 6% Eltesel SXS 40. Both compositions showed the same

high gelling behaviour.

Example 1.11 - STP

[0111] Composition 2 was prepared without polymer (K) and with 1% of the Narlex polymer of Example 1.1 (17). Composition 17 showed very low gelling compared with composition K.

Example 1.12 - Sodium citrate & Dobanol 25-3

[0112] Two compositions (L and 18) were prepared with 10g Dobanol 25-3 mixed with 2g Sodium citrate. The nonionic of composition 18 additionally contained 2% of the Narlex polymer. Composition 18 showed less gelling in the gelling test than composition L.

Example 1.13 - Magnesium Sulphate & Dobanol 25-3

[0113] Two compositions (M and 19) were prepared with 10g Dobanol 25-3 mixed with 2g Magnesium Sulphate. The nonionic of composition 19 additionally contained 2% of the Narlex polymer. Composition 19 showed less gelling in the gelling test than composition M.

Example 1.12 - Sodium EDTA & Dobanol 25-3

[0114] Two compositions (N and 20) were prepared with 10g Dobanol 25-3 mixed with 2g Sodium EDTA. The nonionic of composition 20 additionally contained 2% of the Narlex polymer. Composition 20 showed less gelling in the gelling test than composition N.

Example 2 - dissolution test

[0115] The following compositions were made by adding the ingredients in the order listed. After colloid milling, the compositions were ball-milled after addition of perborate/metasilicate. The resulting compositions have a D(3,2) particle size of from 1-15µm.

Composition 21	(in % by weight)
Nonionic mix 1)	46.4
LAS acid	7.0
Sodium Carbonate	21.0
Calcium Carbonate	7.0
Hydrophobed silica	4.2
Minors	1.7
Perborate	12.2

1) Vista 1012-6.2 and Dobanol 25-3 in 55/45 weight ratio

Composition 22	(in % by weight)
Nonionic mix 1)	40.0

(continued)

Composition 22	(in % by weight)
LAS acid	3.1
Minors	10.9
Sodium metasilicate	46.0

Composition 23	(in % by weight)
Nonionic mix 1)	39.8
Sodium carbonate	18.0
LAS acid	6.0
Calcium carbonate	6.0
Hydrophobed silica	4.0
Sodium perborate	10.5
Solvent	10.0
Minors	5.7

Dissolution test

[0116] 1 gram of product was put in a 100ml beaker that was put in a large 3 liter beaker filled with 2.5 litre water of 20°C and magnetically stirred at 350 rpm. The conductivity (in %) of the water was determined over time.

Example 2.1

[0117] Three compositions (0, 24 and 25) were prepared according to composition 21 with respectively 0, 1 and 2% of the Narlex polymer. Composition 25 showed the best dissolution followed by composition 24.

Example 2.2

[0118] Three compositions (P, 26 and 27) were prepared according to composition 22 with respectively 0, 1 and 2% of the Narlex polymer. Composition 27 showed the best dissolution followed by composition 26.

Example 2.3

[0119] Composition 23 was prepared and 25% by weight of water was added, whereafter the composition was stirred homogeneously. The composition was prepared without polymer (Q) and with 1% of the Narlex polymer of Example 1.1 (Composition 28). Both compositions were tested in the above dissolution test. After 4 minutes, 83% of Composition 28 was dissolved, whereas only 47% of composition Q was dissolved.

Example 3 - Dispersion test

[0120] 1 gram of product (a spot of about 4 cm diameter) was put on a standard CSG test cloth. The cloth

was wetted and mounted on a sample jar that was put in a 3 litre beaker containing 2.5 liters of water of 20°C. About 3 cm above the cloth a six blade stirrer (12 cm diameter) was placed that stirred with 70rpm. The time for complete dispersion of the product from the test cloth was determined.

Example 3.1

[0121] Composition 21 was prepared without polymer (R), with 1 % Narlex polymer (29) and with 2% of the Narlex polymer (30). Composition R was dispersed after 37 minutes, whereas composition 29 was dispersed after 23 minutes and composition 30 after 13 minutes.

Example 3.2

[0122] Composition 21 with 10% additional nonionic mix was prepared without polymer (S) and with 1 % of Narlex polymer (31). Composition S was dispersed after 35 minutes, whereas composition 31 was dispersed after 16 minutes.

Example 3.3

[0123] Composition 23 was prepared without polymer (T) and with 1 % Narlex polymer (32). Composition T was dispersed after 29 minutes, whereas composition 32 was dispersed after 22 minutes.

Example 4 - Dispensing test

[0124] Composition 22 was prepared without polymer (U) and with 1% additional Narlex polymer (33). Composition 33 dispenses better in a system involving three-way valve as disclosed in the figure of EP-A-480,490 than composition U.

Claims

1. A non-aqueous liquid detergent composition comprising a non-aqueous liquid phase which comprises from 10% to 90% by weight of the composition of liquid nonionic surfactant, wherein the composition comprises a polymeric hydrotrope comprising hydrophilic and hydrophobic monomers, wherein the hydrophilic monomer is acrylic acid or methacrylic acid, and wherein the hydrophobic monomer is styrene, wherein the composition further comprises an electrolyte material.
2. A composition according to claim 1 wherein the molar ratio between the hydrophilic and the hydrophobic monomers in the polymer is from 5:1 to 1:5.
3. A composition according to any preceding claim, further comprising solid dispersed particles.

4. A method of preparing a non-aqueous detergent composition according to any preceding claim, wherein the polymeric hydrotrope is added to the composition in a form comprising 25 % or less by weight of water.

Patentansprüche

1. Nicht-wässrige flüssige Waschmittelzusammensetzung, enthaltend eine nicht-wässrige flüssige Phase, die flüssiges nichtionisches Tensid in einer Menge von 10 bis 90 Gew.-% der Zusammensetzung enthält, worin die Zusammensetzung ein polymeres Hydrotrop enthält, umfassend hydrophile und hydrophobe Monomere, worin das hydrophile Monomere Acrylsäure oder Methacrylsäure ist, und worin das hydrophobe Monomere Styrol ist, worin die Zusammensetzung ferner ein Elektrolyt-Material umfasst.
2. Zusammensetzung nach Anspruch 1, worin das molare Verhältnis zwischen hydrophilen und hydrophoben Monomeren in dem Polymeren von 5 : 1 bis 1 : 5 beträgt.
3. Zusammensetzung gemäß irgendeinem vorstehenden Anspruch, ferner feste dispergierte Teilchen enthaltend.
4. Verfahren zur Herstellung einer nicht-wässrigen Waschmittelzusammensetzung gemäß irgendeinem vorstehenden Anspruch, worin das polymere Hydrotrop zu der Zusammensetzung in einer Form zugesetzt wird, enthaltend 25 Gew.-% oder weniger an Wasser.

Revendications

1. Composition détergente liquide non aqueuse comprenant une phase liquide non aqueuse, qui comprend de 10 % à 90 % en poids de la composition de tensioactif non ionique liquide, dans laquelle la composition comprend un hydrotrope polymère comprenant des monomères hydrophiles et hydrophobes, le monomère hydrophile étant l'acide acrylique ou l'acide méthacrylique et le monomère hydrophobe étant le styrène, dans laquelle la composition comprend en plus un électrolyte.
2. Composition selon la revendication 1, dans laquelle le rapport molaire entre les monomères hydrophiles et les monomères hydrophobes dans le polymère est de 5:1 à 1:5.
3. Composition selon l'une quelconque des revendications précédentes comprenant en plus des parti-

cules solides dispersées.

4. Procédé de préparation d'une composition détergente non aqueuse selon l'une quelconque des revendications précédentes, dans lequel l'hydrotrope polymère est ajouté à la composition sous une forme comprenant 25 % ou moins en poids d'eau.

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