(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) **EP 3 517 596 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:
20.03.2024 Bulletin 2024/12

(21) Application number: **18214163.0**

(22) Date of filing: **19.12.2018**

(51) International Patent Classification (IPC):
C11D 3/37 *(2006.01)* ***C11D 3/43*** *(2006.01)*
C11D 17/04 *(2006.01)*

(52) Cooperative Patent Classification (CPC):
C11D 3/3715; C11D 3/3788; C11D 3/43;
C11D 17/043

(54) **METHOD OF MAKING AN OPAQUE LIQUID DETERGENT COMPOSITION**

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PROCÉDÉ DE FABRICATION D'UNE COMPOSITION DÉTERGENTE LIQUIDE OPAQUE

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR

(30) Priority: **25.01.2018 EP 18153446**

(43) Date of publication of application:
31.07.2019 Bulletin 2019/31

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Description

FIELD OF THE INVENTION

[0001] The present invention relates to a method of making an opaque liquid detergent composition.

BACKGROUND OF THE INVENTION

[0002] Liquid laundry detergent compositions are known. There is a tendency today to make liquid detergent compositions more compact and minimize unnecessary 'bulking' ingredients or water. This approach is more environmentally friendly as it reduces carbon footprints from transportation and means less packaging can be used. It is also especially beneficial for water-soluble unit dose articles containing the detergents as said water-soluble unit dose articles have limited space for formulating detergent ingredients.

[0003] The aesthetic appearance of a laundry detergent composition is important in terms of consumer acceptance. Consumers believe opaque detergent compositions are more effective in terms of performance than translucent ones. This is because consumers believe opaque detergent compositions are more concentrated and so more effective.

[0004] Often, opacifying agents need to be added to the liquid laundry detergent composition to make it opaque; these opacifying agents do not add any fabric cleaning or treatment performance value. However, compacted liquid laundry detergent compositions have limited space for addition of such ingredients, especially considering these actives typically are formulated as low active slurries. If they are added, this may require the reduction in the concentration of another cleaning ingredients which would affect overall cleaning performance of the liquid detergent composition.

[0005] Therefore, there is a need in the art for a method to make an opaque liquid laundry detergent composition without the addition of specific non-performance value adding opacifying materials.

[0006] It was surprisingly found that the process according to the present invention, using a specific polymer premix, overcame this issue. The polymer premix comprises an anionic polyester terephthalate polymer, which provides soil release benefits on fabrics being laundered, and other common laundry detergent ingredients. The process further involves the use of a surfactant premix. The surfactant premix also comprises common detergent ingredients necessary to provide cleaning benefits. However, the specific method for formulating these ingredients provided an opaque liquid laundry detergent composition. Hence an opaque liquid laundry detergent composition was achieved using common laundry detergent ingredients and without the addition of specific non-performance value adding opacifying materials.

[0007] US2017292089A, US2015005005220A and WO2017133879A disclose methods of manufacturing liquid detergent compositions comprising the steps of preparing a liquid detergent composition comprising surfactant and non-aqueous solvent, and adding a polyester terephthalate to this. They do not disclose a polymer premix as according to the present invention.

SUMMARY OF THE INVENTION

[0008] A first aspect of the present invention is a method of making an opaque liquid detergent composition comprising the steps of;

a. preparing a polymer premix, wherein the polymer premix comprises

i. between 5% and 40% by weight of the polymer premix of an anionic polyester terephthalate polymer, wherein the anionic polyester terephthalate polymer has a polyester terephthalate backbone grafted with one or more anionic groups;
ii. between 60% and 95% by weight of the polymer premix of a solvent, wherein the solvent is an aqueous solvent, a non-aqueous solvent or a mixture thereof;

b. preparing a surfactant premix, wherein the surfactant premix comprises between 10% and 70%, preferably between 20% and 65%, more preferably between 40% and 60% by weight of the surfactant premix of a non-soap surfactant, the non-soap surfactant preferably comprising between 50% and 100% preferably between 70% and 100% more preferably between 90% and 100% by weight of the non-soap surfactant of a non-soap anionic surfactant, the non-soap anionic surfactant comprising the protonated form of the non-soap anionic surfactant, the neutralized salt form of a non-soap anionic surfactant or a mixture thereof, preferably wherein the non-soap anionic surfactant premix comprises between 50% and 90% by weight of the non-soap anionic surfactant of the neutralized salt form of a non-soap anionic surfactant;
c. combining the polymer premix and the surfactant premix in a weight ratio of the polymer premix to the surfactant premix of 2:1 and 1:25, preferably between 1.5:1 and 1:10;

d. mixing the polymer premix and the surfactant premix;
e. Optionally adding one or more adjunct ingredients;
f. Collecting the final opaque liquid detergent composition,

wherein, by 'opaque' it is meant a liquid composition having less than 1% transmittance measured using a ColorQuest XE spectrophotometer, using 2.5 mL PS cuvettes (1 cm path length), measuring a range from 400 to 700 nm when measured neat.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009] FIG. 1 is a water-soluble unit dose article made according to the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Method

[0010] An aspect of the present invention is a method of making an opaque liquid laundry detergent composition. The term 'liquid laundry detergent composition' refers to any laundry detergent composition comprising a liquid capable of wetting and treating a fabric, and includes, but is not limited to, liquids, gels, pastes, dispersions and the like. The liquid composition can include solids or gases in suitably subdivided form, but the liquid composition excludes forms which are non-fluid overall, such as tablets or granules.

[0011] By 'opaque' we herein mean a liquid composition having less than 1% transmittance measured using a ColorQuest XE spectrophotometer, using 2.5 mL PS cuvettes (1 cm path length), measuring a range from 400 to 700 nm when measured neat.

[0012] The method comprises the steps of;

a. Preparing a polymer premix, wherein the polymer premix comprises

i. between 5% and 40% by weight of the polymer premix of an anionic polyester terephthalate polymer, wherein the anionic polyester terephthalate polymer is a polyester terephthalate backbone grafted with one or more anionic groups;
ii. between 60% and 95% by weight of the polymer premix of a solvent, wherein the solvent is an aqueous solvent, a non-aqueous solvent or a mixture thereof.

[0013] Preferably, the polymer premix is prepared at a temperature above the glass transition temperature of the polyester terephthalate. Those skilled in the art will be aware of how to calculate the glass transition temperature and how to adjust and maintain the temperature above the glass transition temperature of the polyester terephthalate. Preferably, the polymer premix is prepared at a temperature of between 50°C and 80°C, more preferably between 50°C and 70°C, even more preferably between 50°C and 65°C.

[0014] The anionic polyester terephthalate polymer and the solvent may be added to one another in any order. For example, the anionic polyester terephthalate polymer may be added to the solvent. Alternatively, the solvent may be added to the anionic polyester terephthalate polymer.

[0015] The anionic polyester terephthalate polymer and the polymer may be mixed using any suitable mixer.

[0016] Those skilled in the art will be aware of suitable mixing means. Suitable mixers include static mixers, dynamic mixers or a combination thereof. When mixing with a static mixer, preferably the solvent and anionic polyester terephthalate polymer are mixed together between 1/s and 100/s, more preferably between 3/s and 10/s at a temperature of between 15°C and 30°C, more preferably between 17°C and 25°C. Examples of suitable static mixers include the SMX range of static mixers commercially available from Sulzer. When using a dynamic mixer, preferably the solvent and anionic polyester terephthalate polymer are mixed together between 40rpm and 500rpm. Preferably, the anionic polyester terephthalate polymer and the solvent are mixed for between 1 minutes and 20 minutes, preferably between 2.5 minutes and 18 minutes using the dynamic mixer at a temperature of between 15°C and 30°C, more preferably between 17°C and 25°C. Examples of suitable dynamic mixers include include pitch blade mixers, disc turbine mixers or propeller mixers. Such mixers are well known and available from numerous commercial sources.

[0017] The polymer premix is described in more detail below.

b. Preparing a surfactant premix, wherein the surfactant premix comprises between 10% and 70%, preferably between 20% and 65%, more preferably between 40% and 60% by weight of the surfactant premix of a non-soap surfactant, the non-soap surfactant preferably comprising between 50% and 100%, more preferably between 70% and 100%, even more preferably between 90% and 100% by weight of the non-soap surfactant of a non-soap anionic surfactant, the

non-soap anionic surfactant comprising the protonated form of the non-soap anionic surfactant, the neutralized salt form of a non-soap anionic surfactant or a mixture thereof, preferably wherein the non-soap anionic surfactant premix comprises between 50% and 90% by weight of the non-soap anionic surfactant of the neutralized salt form of a non-soap anionic surfactant.

[0018] The surfactant premix is described in more detail below.

c. Combining the polymer premix and the surfactant premix in a weight ratio of the polymer premix to the surfactant premix of 2:1 and 1:25, preferably between 1.5:1 and 1:10.

[0019] The polymer premix and the surfactant premix may be added to one another in any order. For example, the polymer premix may be added to the surfactant premix. Alternatively, the surfactant premix may be added to the polymer premix. Preferably, the polymer premix is added to the surfactant premix. Preferably, the surfactant premix is being mixed as the polymer premix is added to ensure the polymer premix is efficiently mixed into the surfactant premix as they are combined.

[0020] The surfactant premix can be mixed using any suitable mixing means. Those skilled in the art will be aware of suitable mixers. Suitable mixers include static mixers, dynamic mixers or a combination thereof. When mixing with a static mixer, preferably the surfactant premix is mixed between 1/s and 100/s, more preferably between 3/s and 10/s at a temperature of between 15°C and 30°C, more preferably between 17°C and 25°C. Examples of suitable static mixers include the SMX range of static mixers commercially available from Sulzer. When using a dynamic mixer, preferably the surfactant premix is mixed between 40rpm and 500rpm. Examples of suitable dynamic mixers include include pitch blade mixers, disc turbine mixers or propeller mixers. Such mixers are well known and available from numerous commercial sources.

[0021] Preferably, the polymer premix and the surfactant premix are combined at a temperature of between 15°C and 30°C, more preferably between 17°C and 25°C.

d. Mixing the polymer premix and the surfactant premix.

[0022] Those skilled in the art will be aware of suitable mixing means. Suitable mixers include static mixers, dynamic mixers or a combination thereof. When mixing with a static mixer, preferably the surfactant premix and polymer premix are mixed together between 1/s and 100/s, more preferably between 3/s and 10/s at a temperature of between 15°C and 30°C, more preferably between 17°C and 25°C. Examples of suitable static mixers include the SMX range of static mixers commercially available from Sulzer. When using a dynamic mixer, preferably the surfactant premix and polymer premix are mixed together between 40rpm and 500rpm. Preferably, the polymer premix and the surfactant premix are mixed for between 1 minutes and 20 minutes, preferably between 2.5 minutes and 18 minutes using the dynamic mixer at a temperature of between 15°C and 30°C, more preferably between 17°C and 25°C. Examples of suitable dynamic mixers include include pitch blade mixers, disc turbine mixers or propeller mixers. Such mixers are well known and available from numerous commercial sources.

e. Optionally adding one or more adjunct ingredients.

[0023] The adjunct ingredient may be selected from non-ionic surfactants, chelants, perfumes, dyes, enzymes, polyethyleneimines, polysaccharide polymers, polyethylene glycol polymers or a mixture thereof. The liquid laundry detergent composition may further comprise other adjunct ingredient selected from hueing dyes, polymers, builders, dye transfer inhibiting agents, dispersants, enzyme stabilizers, catalytic materials, bleach, bleach activators, polymeric dispersing agents, anti-redeposition agents, suds suppressors, aesthetic dyes, opacifiers, perfumes, perfume delivery systems, structurants, hydrotropes, processing aids, pigments, amphoteric surfactants, cyclic diamines, zwitterionic polyamines, anti-oxidants, preservatives and mixtures thereof. The adjunct ingredients may be added in any order. Those skilled the art will be aware of how to add the adjunct ingredient(s) and at what concentration.

f. Collecting the final opaque liquid detergent composition.

[0024] Those skilled in the art will be aware of how to collect the final opaque liquid laundry detergent composition. The liquid laundry detergent composition may be collected in a tank or container for intermediate storage. Alternative, the liquid laundry detergent composition may be collected into a bottle wherein the bottle may be sold directly to a consumer. Alternatively, the liquid laundry detergent composition may be collected into a water-soluble unit dose article. The liquid laundry detergent composition may be temporarily stored in a storage tank ahead of being added to a water-soluble unit dose article. The water-soluble unit dose article comprises the water-soluble film shaped such that the unit-dose article comprises at least one internal compartment surrounded by the water-soluble film. The unit dose article may comprise a first water-soluble film and a second water-soluble film sealed to one another such to define the internal compartment. The water-soluble unit dose article is constructed such that the detergent composition does not leak out

of the compartment during storage. However, upon addition of the water-soluble unit dose article to water, the water-soluble film dissolves and releases the contents of the internal compartment into the wash liquor.

[0025] The compartment should be understood as meaning a closed internal space within the unit dose article, which holds the detergent composition. During manufacture, a first water-soluble film may be shaped to comprise an open compartment into which the detergent composition is added. A second water-soluble film is then laid over the first film in such an orientation as to close the opening of the compartment. The first and second films are then sealed together along a seal region.

[0026] The unit dose article may comprise more than one compartment, even at least two compartments, or even at least three compartments. The compartments may be arranged in superposed orientation, i.e. one positioned on top of the other. In such an orientation the unit dose article will comprise three films, top, middle and bottom. Alternatively, the compartments may be positioned in a side-by-side orientation, i.e. one orientated next to the other. The compartments may even be orientated in a 'tyre and rim' arrangement, i.e. a first compartment is positioned next to a second compartment, but the first compartment at least partially surrounds the second compartment, but does not completely enclose the second compartment. Alternatively one compartment may be completely enclosed within another compartment.

[0027] Wherein the unit dose article comprises at least two compartments, one of the compartments may be smaller than the other compartment. Wherein the unit dose article comprises at least three compartments, two of the compartments may be smaller than the third compartment, and preferably the smaller compartments are superposed on the larger compartment. The superposed compartments preferably are orientated side-by-side.

[0028] In a multi-compartment orientation, the detergent composition according to the present invention may be comprised in at least one of the compartments. It may for example be comprised in just one compartment, or may be comprised in two compartments, or even in three compartments.

[0029] Each compartment may comprise the same or different compositions. The different compositions could all be in the same form, or they may be in different forms.

The water-soluble unit dose article may comprise at least two internal compartments, wherein the liquid laundry detergent composition is comprised in at least one of the compartments, preferably wherein the unit dose article comprises at least three compartments, wherein the detergent composition is comprised in at least one of the compartments.

[0030] FIG. 1 discloses a water-soluble unit dose article (1) according to the present invention. The water-soluble unit dose article (1) comprises a first water-soluble film (2) and a second water-soluble film (3) which are sealed together at a seal region (4). The liquid laundry detergent composition (5) is comprised within the water-soluble soluble unit dose article (1).

[0031] The film of the present invention is soluble or dispersible in water. The water-soluble film preferably has a thickness of from 20 to 150 micron, preferably 35 to 125 micron, even more preferably 50 to 110 micron, most preferably about 76 micron.

[0032] Preferably, the film has a water-solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out here after using a glass-filter with a maximum pore size of 20 microns:

5 grams ±0.1 gram of film material is added in a pre-weighed 3L beaker and 2L ± 5ml of distilled water is added. This is stirred vigorously on a magnetic stirrer, Labline model No. 1250 or equivalent and 5 cm magnetic stirrer, set at 600 rpm, for 30 minutes at 30°C. Then, the mixture is filtered through a folded qualitative sintered-glass filter with a pore size as defined above (max. 20 micron). The water is dried off from the collected filtrate by any conventional method, and the weight of the remaining material is determined (which is the dissolved or dispersed fraction). Then, the percentage solubility or dispersability can be calculated.

[0033] Preferred film materials are preferably polymeric materials. The film material can, for example, be obtained by casting, blow-moulding, extrusion or blown extrusion of the polymeric material, as known in the art.

[0034] Preferred polymers, copolymers or derivatives thereof suitable for use as pouch material are selected from polyvinyl alcohols, polyvinyl pyrrolidone, polyalkylene oxides, acrylamide, acrylic acid, cellulose, cellulose ethers, cellulose esters, cellulose amides, polyvinyl acetates, polycarboxylic acids and salts, polyaminoacids or peptides, polyamides, polyacrylamide, copolymers of maleic/acrylic acids, polysaccharides including starch and gelatine, natural gums such as xanthum and carragum. More preferred polymers are selected from polyacrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polymethacrylates, and most preferably selected from polyvinyl alcohols, polyvinyl alcohol copolymers and hydroxypropyl methyl cellulose (HPMC), and combinations thereof. Preferably, the level of polymer in the pouch material, for example a PVA polymer, is at least 60%. The polymer can have any weight average molecular weight, preferably from about 1000 to 1,000,000, more preferably from about 10,000 to 300,000 yet more preferably from about 20,000 to 150,000.

[0035] Preferably, the water-soluble film comprises polyvinyl alcohol polymer or copolymer, preferably a blend of polyvinylalcohol polymers and/or polyvinylalcohol copolymers, preferably selected from sulphonated and carboxylated anionic polyvinylalcohol copolymers especially carboxylated anionic polyvinylalcohol copolymers, most preferably a blend of a polyvinylalcohol homopolymer and a carboxylated anionic polyvinylalcohol copolymer.

[0036] Preferred films exhibit good dissolution in cold water, meaning unheated distilled water. Preferably such films exhibit good dissolution at temperatures of 24°C, even more preferably at 10°C. By good dissolution it is meant that the film exhibits water-solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out here after using a glass-filter with a maximum pore size of 20 microns, described above.

[0037] Preferred films are those supplied by Monosol under the trade references M8630, M8900, M8779, M8310.

[0038] The film may be opaque, transparent or translucent. The film may comprise a printed area.

[0039] The area of print may be achieved using standard techniques, such as flexographic printing or inkjet printing.

[0040] The film may comprise an aversive agent, for example a bittering agent. Suitable bittering agents include, but are not limited to, naringin, sucrose octaacetate, quinine hydrochloride, denatonium benzoate, or mixtures thereof. Any suitable level of aversive agent may be used in the film. Suitable levels include, but are not limited to, 1 to 5000ppm, or even 100 to 2500ppm, or even 250 to 2000rpm.

[0041] The method may be a continuous method or a batch method.

Polymer premix

[0042] The polymer premix comprises between 5% and 40%, preferably between 10% and 35%, more preferably between 15% and 25% by weight of the polymer composition of a polyester terephthalate, wherein the polyester terephthalate is a polyester terephthalate backbone grafted with one or more anionic groups. The polyester terephthalate is described in more detail below.

[0043] Without wishing to be bound by theory, if the level of the polyester terephthalate is too low, then sub-optimal opacifying effect is achieved in the final liquid laundry detergent composition. If the level of polyester terephthalate is too high in the polymer composition, then it does not dissolve and results in undissolved sediments present in the polymer premix and the liquid laundry detergent composition. To dissolve excess undissolved polyester terephthalate, addition of more water or non-aqueous solvent is needed, but this then brings further non-performance value adding materials.

[0044] Therefore, the present invention also addresses the problem of maximizing the level of polyester terephthalate that can be effectively formulated and that achieves effective opacity of the final liquid laundry detergent composition whilst maintaining stability of the polymer premix and the final liquid laundry detergent composition.

[0045] The polymer premix comprises between 5% and 40%, preferably between 5% and 15%, more preferably between 6% and 12%, even more preferably between 7% and 10% by weight of the polymer composition of water.

[0046] The polymer premix comprises between 20% and 90%, preferably between 55% and 85%, more preferably between 60% and 80%, most preferably between 65% and 75% by weight of the polymer composition of a non-aqueous solvent. Preferably the non-aqueous solvent and the water are in a weight ratio of 5:1 to 15:1, preferably from 8:1 to 10:1.

[0047] Without wishing to be bound by theory, it is especially preferred to control the levels and ratio of non-aqueous solvent and water when the polymer premix will be used to formulate into a liquid laundry detergent composition within a water-soluble unit dose article. Such control is necessary to ensure integrity of the water-soluble film. Too much water will cause failure of the film due to premature dissolution, and too much non-aqueous solvent will cause failure of the water-soluble unit dose article due to the film being too 'floppy' and the unit dose article losing structural integrity.

[0048] Preferably, the polymer premix comprises between 20% and 90%, more preferably between 55% and 85%, even more preferably between 60% and 80%, most preferably between 65% and 75% by weight of the polymer composition of a non-aqueous solvent wherein the non-aqueous solvent comprises ethanol, propanol preferably 1-propanol, butanol preferably 1-butanol, ethylene glycol, propylene glycol preferably 1,2-propylene glycol, dipropylene glycol, tripropylene glycol, polyethylene glycol, polypropylene glycol, glycerol, trimethylene glycol, or a mixture thereof, wherein preferably the polyethylene glycol, polypropylene glycol or mixture thereof has an average molecular weight between 100 and 800 more preferably from 200 to 400. More preferably, the polymer premix comprises between 20% and 90%, more preferably between 55% and 85%, even more preferably between 60% and 80%, most preferably between 65% and 75% by weight of the polymer premix of a non-aqueous solvent wherein the non-aqueous solvent is selected from dipropyleneglycol, tripropyleneglycol, propylene glycol preferably 1,2-propylene glycol and glycerol and a mixture thereof, most preferably a mixture of propylene glycol preferably 1,2-propyleneglycol and glycerol. Most preferably the propylene glycol and the glycerol are in a weight ratio of from 2:1 to 5:1, preferably from 3:1 to 4:1.

[0049] Preferably the polymer premix comprises from 60% to 95% preferably from 65% to 90% more preferably from 75% to 85% by weight of the polymer composition of aqueous and non-aqueous solvent. Preferably the polymer premix comprises from 5% to 15% of water, from 15% to 25% of glycerol and from 60% to 80% of propylene glycol preferably 1,2-propylene glycol, by weight of the polymer combined aqueous and non-aqueous solvent composition.

[0050] Preferably, the polymer premix has a pH of between 3 and 8, preferably between 4 and 6, measured neat at 20°. Those skilled in the art will be aware of known methods to measure the pH. Those skilled in the art will be aware of how to adjust the pH to be within the preferred range by the addition of suitable acidic or alkali materials.

[0051] Preferably, the polymer premix has a viscosity of between 10 mPa.s and 1000 mPa.s, preferably between 100 mPa.s and 750 mPa.s, most preferably between 200 mPa.s and 400 mPa.s at 20 °C when measured using a TA

Instruments rheometer (preferably model AR-G2, AR2000ex or equivalent) at 10/s with a 40mm parallel plate & 500 or 1000 μm gap wherein the procedure including a conditioning step, 30s at 20°C with no pre-shear followed by continuous ramp from 0.01 to 1200 /s, log scale, 32 points per decade, 3 minutes run.

[0052] The polymer premix is opaque. By 'opaque' we herein mean a liquid composition having less than 1% transmittance measured using a ColorQuest XE spectrophotometer, using 2.5 mL PS cuvettes (1 cm path length), measuring a range from 400 to 700 nm when measured neat.

Surfactant premix

[0053] The surfactant premix comprises between 10% and 70%, preferably between 20% and 65%, more preferably between 40% and 60% by weight of the surfactant premix of a non-soap surfactant, the non-soap surfactant preferably comprising between 50% and 100% preferably between 70% and 100% more preferably between 90% and 100% by weight of the non-soap surfactant of a non-soap anionic surfactant, the non-soap anionic surfactant comprising the protonated form of the non-soap anionic surfactant, the neutralized salt form of a non-soap anionic surfactant or a mixture thereof, preferably wherein the non-soap anionic surfactant premix comprises between 50% and 90% preferably between 55% and 85% by weight of the non-soap anionic surfactant of the neutralized salt form of a non-soap anionic surfactant, and between 10% and 50%, preferably between 15% and 45% by weight of the surfactant premix of the acid form of a non-soap anionic surfactant. Those skilled in the art will know how to neutralize the acid form of the non-soap anionic surfactant using suitable neutralizing agents. The surfactant premix may comprise monoethanolamine, triethanolamine, an alkali metal or a mixture thereof.

[0054] Preferably, the non-soap anionic surfactant is selected from an alkyl benzene sulphonate, an alkyl sulphate, an alkoxylated alkyl sulphate or a mixture thereof.

[0055] More preferably, the non-soap anionic surfactant is a mixture of linear alkylbenzene sulphonate and alkoxylated alkyl sulphate, more preferably a mixture of linear alkylbenzene sulphonate and ethoxylated alkyl sulphate.

[0056] Preferably, the weight ratio of linear alkylbenzene sulphonate to alkoxylated alkyl sulphate, more preferably linear alkylbenzene sulphonate to ethoxylated alkyl sulphate is from 1:2 to 20:1, preferably from 1.1:1 to 15:1, more preferably from 1.2:1 to 10:1, even more preferably from 1.3:1 to 5:1, most preferably from 1.4:1 to 3:1.

[0057] The weight ratio of linear alkylbenzene sulphonate to ethoxylated alkyl sulphate may be from 1:10 to 20:1, preferably from 1:7 to 3:1, more preferably from 1:5 to 1.5:1.

[0058] Preferably the liquid laundry detergent composition may comprise up to 50%, preferably between 5% and 50%, more preferably between 10% and 45%, even more preferably between 15% and 40% by weight of the liquid laundry detergent composition of a non-soap anionic surfactant.

[0059] The surfactant premix may comprise a solvent and wherein the solvent comprised in the surfactant premix may be selected from water, ethanol, propanol preferably 1-propanol, butanol preferably 1-butanol, ethylene glycol, propylene glycol preferably 1,2-propylene glycol dipropylene glycol, tripropyleneglycol, polyethyleneglycol or polypropyleneglycol preferably with an average molecular weight between 100 and 800 preferably 200 and 400, glycerol, trimethylene glycol, or a mixture thereof, preferably water, propylene glycol, dipropylene glycol, tripropylene glycol, glycerol and a mixture thereof.

The liquid laundry detergent composition

[0060] Preferably the liquid detergent further comprises nonionic surfactant. This nonionic surfactant may be formulated as part of the surfactant premix or may be post-added, preferably as part of the surfactant premix. The liquid laundry detergent composition preferably comprises less than 10%, preferably between 0% and 9.5%, preferably between 0.01% and 9%, more preferably between 0.1% and 7%, even more preferably between 1% and 5%, most preferably between 1 and 3% by weight of the liquid laundry detergent composition of a fatty alcohol ethoxylate non-ionic surfactant. Preferably, the fatty alcohol ethoxylate has an average alkyl carbon chain length of between 5 and 30, preferably between 8 and 18, more preferably between 10 and 16, most preferably between 12 and 15. Preferably, the fatty alcohol ethoxylate has an average degree of ethoxylation of between 0.5 and 20, preferably between 1 and 15, more preferably between 5 and 12, even more preferably between 6 and 10, most preferably between 7 and 8.

[0061] Preferably, the weight ratio of non-soap anionic surfactant to fatty alcohol ethoxylate non-ionic surfactant is between 5:1 and 23:1 preferably between 7:1 and 23:1, more preferably between 8:1 and 23:1, most preferably between 9:1 and 20:1.

[0062] The liquid laundry detergent composition may comprise less than 10%, preferably less than 8%, most preferably between 1% and 8% by weight of the liquid laundry detergent composition of fatty acid, neutralised fatty acid soap or a mixture thereof. This fatty acid, neutralized fatty acid soap or mixtures thereof may be formulated as part of the surfactant premix or may be post-added, preferably as part of the surfactant premix.

[0063] The solvent comprised in the liquid detergent composition may be selected from water, ethanol, propanol

preferably 1-propanol, butanol preferably 1-butanol, ethylene glycol, propylene glycol preferably 1,2-propylene glycol, dipropylene glycol, tripropylene glycol, polyethyleneglycol or polypropyleneglycol preferably with an average molecular weight between 100 and 800 preferably 200 and 400, glycerol, trimethylene glycol, or a mixture thereof, preferably water, propylene glycol, dipropylene glycol, tripropylene glycol, glycerol and a mixture thereof. Preferably the solvent comprises from 10% to 40% preferably from 15% to 30% by weight of the liquid detergent composition of a non-aqueous solvent, the non-aqueous solvent preferably comprising from 40% to 80%, preferably from 50% to 70% by weight of the non-aqueous solvent of the propylene glycol, from 10% to 30%, preferably from 15% to 25% by weight of the non-aqueous solvent of dipropylene glycol, tri-propylene glycol and a mixture thereof, and from 10% to 30%, preferably from 15% to 25% by weight of the non-aqueous solvent of glycerol. Preferably the liquid detergent composition will comprise between 1 and 25% preferably between 5% and 15% by weight of the final composition of water.

[0064] The liquid laundry detergent composition may also comprise adjunct ingredients which may be selected from chelants, perfumes, dyes, enzymes, (alkoxylated) polyethyleneimines, polysaccharide polymers, polyethylene glycol polymers or a mixture thereof. The liquid laundry detergent composition may further comprise other adjunct ingredient selected from hueing dyes, polymers, builders, dye transfer inhibiting agents, dispersants, enzyme stabilizers, catalytic materials, bleach, bleach activators, polymeric dispersing agents, anti-redeposition agents, suds suppressors, aesthetic dyes, opacifiers, perfumes, perfume delivery systems, structurants, hydrotropes, processing aids, pigments, amphoteric surfactants, cyclic diamines, zwitterionic polyamines, anti-oxidants, preservatives and mixtures thereof.

[0065] Preferably, the liquid laundry detergent composition has a pH from 6 to 10 preferably from 7 to 9, more preferably from 7 to 8. Preferably, the liquid laundry detergent composition comprises a pH adjusting agent selected from alkanolamines, preferably monethanolamine, diethanolamine, triethanolamine or a mixture thereof, most preferably monoethanolamine.

Polyester terephthalate

[0066] The polyester terephthalate is a polyester terephthalate backbone grafted with one or more anionic groups, more preferably, an anionic polyester of propylene terephtalate.

[0067] Suitable anionic polyesters are those that are derived from terephtalic acid, 5-sulfoisophtalic acid or the salt of 5-sulfoisophtalic acid, from ethylene glycol or polyethylene glycol, propylene glycol or polypropylene glycol and polyalkyleneglycol monoalkyl ether, and optionally from further monomers having 3 to 6 functions capable of polycondensation, in particular acid, alcohol or ester functions.

[0068] Preferably, the polyester terephthalate comprises the combination of structural units (I) to (III):

(I) $-[(OCHR1\text{-}CHR^2)_a\text{-}O\text{-}OC\text{-}Ar\text{-}CO\text{-}]_d$

(II) $-[(OCHR^3\text{-}CHR^4)_b\text{-}O\text{-}OC\text{-}sAr\text{-}CO\text{-}]_e$

(III) $-[(OCHR^5\text{-}CHR^6)_c\text{-}OR^7]_f$

wherein:

a, b and c are from 1 to 200;
d, e and f are from 1 to 50;
Ar is a 1,4-substituted phenylene;
sAr is 1,3-substituted phenylene substituted in position 5 with SO_3Me;
Me is Li, K, Mg/2, Ca/2, Al/3, ammonium, mono-, di-, tri-, or tetraalkylammonium wherein the alkyl groups are (C_1-C_{22}) alkyl or (C_2-C_{10}) hydroxyalkyl, or mixtures thereof;
R^1, R^2, R^3, R^4, R^5 and R^6 are independently selected from H or (Ci-Cis) n- or iso-alkyl preferably methyl; and R^7 is a linear or branched (C_1-C_{18}) alkyl, or a linear or branched (C_2-C_{30}) alkenyl, or a cycloalkyl group with 5 to 9 carbon atoms, a (C_6-C_{30}) aryl group or a (C_6-C_{50}) arylalkyl group preferably phenyl or benzyl.

[0069] Preferably, the polyester terephthalate comprises the structural units (I) to (III) wherein;

R^1 to R^6 independently are H or methyl,
R^7 is methyl,
a, b and c are a number from 1 to 20, preferably a and b are 1 and c is a number from 2 to 10,
d is a number between 1 and 25, preferably between 1 and 10, more preferably between 1 and 5,
e is a number between 1 and 30, preferably between 2 and 15, more preferably between 3 and 10, and
f is a number between 0.05 and 15, preferably between 0.1 and 10, more preferably between 0.25 and 3.

[0070] The polyester therephthalates according to the invention generally have a number average molecular weight in the range of 700 to 50000 g/mol, preferably 800 to 25000 g/mol, more preferably 1000 to 15000 g/mol, most preferably 1200 to 12000 g/mol.

[0071] Suitable soil release polymers are sold by Clariant under the TexCare® series of polymers, e.g. TexCare® SRA300.

[0072] The liquid laundry detergent composition may comprise between 0.1% and 10%, preferably between 0.5% and 8%, more preferably between 1% and 7%, even more preferably between 1.5% and 6%, most preferably between 2% and 6% by weight of the liquid laundry detergent composition of an anionic polyester terephthalates soil release polymer.

[0073] The liquid detergent composition is opaque. By 'opaque' we herein mean a liquid composition having less than 1% transmittance measured using a ColorQuest XE spectrophotometer, using 2.5 mL PS cuvettes (1 cm path length), measuring a range from 400 to 700 nm when measured neat.

[0074] The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40 mm" is intended to mean "about 40 mm."

EXAMPLES

[0075] FIG. 1 discloses a water-soluble unit dose article (1) made according to the present invention. The water-soluble unit dose article (1) comprises a first water-soluble film (2) and a second water-soluble film (3) which are sealed together at a seal region (4). The liquid laundry detergent composition (5) is comprised within the water-soluble soluble unit dose article (1).

Example 1

[0076] A polymer premix according to the invention (Example I) has been created and added to a surfactant premix to demonstrate its ability to opacify a detergent composition. The same polymer has also been directly added as a powder to this surfactant premix as a comparative example I. While addition of the polymer premix according to the invention (Example I) led to the desired opacification of the resulting detergent composition, direct addition of the polymer powder (Comparative Example I) did not equally opacify the detergent composition. Beyond a physically unstable detergent formulation is obtained with the Comparative Example I.

[0077] When formulating a polymer premix comprising more than 40% of Texcare SRA300, i.e. a polymer according to the invention, at equal P-Diol - glycerol - water ratios (Comparative Example II) physical separation of undissolved Texcare SRA300 powder is equally observed after overnight storage.

[0078] Without wishing to be bound by theory it is believed that the dissolved Texcare SRA300 polymers within the polymer premix re-orientate in finely dispersed micron sized droplets, as evidenced by microscopy measurements, providing opacity accordingly, contrary to direct powder added Texcare SRA300 which remains undissolved in the surfactant premix.

[0079] Premixes :

• Anionic polyester terephthalate : Texcare SRA 300 (ex Clariant)

• Polymer premix (according to the invention): The polymer premix is produced through standard mixing of the 1,2-propanediol, glycerol and water in below proportions. Consequently Texcare SRA300 is added as a powder and stirred for 1 hour at 55°C till, after initial observation of powder dissolution during first 15 minutes, an opaque premix has developed. Microscopy confirmed the presence of finely dispersed micron sized droplets while absence of undissolved Texcare SRA300 particles.

Material	Wt%
1,2-Propanediol	56%
Glycerol	16%
Water	8%
Texcare SRA 300	20%

• Surfactant premix : The surfactant premix is produced through standard mixing of the components described :

Material	Wt%
Citric acid	0.87
1,2-propanediol	9.3
Dipropyleneglycol	4.63
monoethanolamine	9.38
glycerol	4.67
HEDP chelant	2.79
K2SO3	0.06
Neodol 24/7	2.52
HLAS	26.69
Brightener 49	0.02
TPK fatty acid	2.01
C1214AE3S	18.11
Ethoxylated polyethyleneimine (PEI600EO20)	4.94
MgCl2	1.27
Water	Balance

• Preparation of a polymer premix - surfactant premix mixture : The polymer premix and surfactant premix are mixed through standard mixing at room temperature such that Texcare SRA300 will be present at 5.8% in the resulting composition.
• Direct addition of Texcare SRA300 powder to the surfactant premix : Texcare SRA300 powder has been added to the surfactant premix such that Texcare SRA300 will be present at 5.8% in the resulting composition. The solution has been mixed through standard mixing at 55°C for 1 hour. Alternatively the Texcare SRA300 has been added at different stages of the surfactant premix making at varying pH (1.2 till 9.7), temperature (up till 93°C) and mixing times (up till 1h30).

Test Results :

[0080] While the polymer premix - surfactant premix mixture (example I) resulted in a physically stable and opaque detergent composition, despite similar processing activities, the addition of Texcare SRA300 directly as a powder onto the surfactant premix or during varying stages of the surfactant premix making (Comparative Example I) did not enable to achieve the same degree of opacity as with the polymer premix according to the invention (Example I) and was still comprising undissolved Texcare SRA300 particles, as evidenced by microscopy. Moreover, phase separation is observed with the Comparative Example I formulation after overnight storage at room temperature, resulting in a clear surfactant premix phase and separated undissolved Texcare SRA300 powder.

Claims

1. A method of making an opaque liquid detergent composition comprising the steps of;

a. preparing a polymer premix, wherein the polymer premix comprises

i. between 5% and 40% by weight of the polymer premix of an anionic polyester terephthalate polymer, wherein the anionic polyester terephthalate polymer has a polyester terephthalate backbone grafted with one or more anionic groups;
ii. between 60% and 95% by weight of the polymer premix of a solvent, wherein the solvent is an aqueous solvent, a non-aqueous solvent or a mixture thereof;

b. preparing a surfactant premix, wherein the surfactant premix comprises between 10% and 70%, preferably between 20% and 65%, more preferably between 40% and 60% by weight of the surfactant premix of a non-

soap surfactant, the non-soap surfactant preferably comprising between 50% and 100% preferably between 70% and 100% more preferably between 90% and 100% by weight of the non-soap surfactant of a non-soap anionic surfactant, the non-soap anionic surfactant comprising the protonated form of the non-soap anionic surfactant, the neutralized salt form of a non-soap anionic surfactant or a mixture thereof, preferably wherein the non-soap anionic surfactant premix comprises between 50% and 90% by weight of the non-soap anionic surfactant of the neutralized salt form of a non-soap anionic surfactant;
c. combining the polymer premix and the surfactant premix in a weight ratio of the polymer premix to the surfactant premix of 2:1 and 1:25, preferably between 1.5:1 and 1:10;
d. mixing the polymer premix and the surfactant premix;
e. Optionally adding one or more adjunct ingredients;
f. Collecting the final opaque liquid detergent composition;

wherein, by 'opaque' it is meant a liquid composition having less than 1% transmittance measured using a ColorQuest XE spectrophotometer, using 2.5 mL PS cuvettes (1 cm path length), measuring a range from 400 to 700 nm when measured neat.

2. The method according to claim 1, wherein the method is a continuous method or a batch method.

3. The method according to any preceding claims wherein the polymer premix has a pH of between 4 and 8, preferably between 5 and 7.

4. The method according to any preceding claims wherein the surfactant premix comprises monoethanolamine, triethanolamine, an alkali metal or a mixture thereof.

5. The methods according to any preceding claims wherein the polyester terephthalate comprises the structural units (I) to (III):

(I) $-[(OCHR1\text{-}CHR^2)_a\text{-}O\text{-}OC\text{-}Ar\text{-}CO\text{-}]_d$

(II) $-[(OCHR^3\text{-}CHR^4)_b\text{-}O\text{-}OC\text{-}sAr\text{-}CO\text{-}]_e$

(III) $-[(OCHR^5\text{-}CHR^6)_c\text{-}OR^7]_f$

wherein:

a, b and c are from 1 to 200;
d, e and f are from 1 to 50;
Ar is a 1,4-substituted phenylene;
sAr is 1,3-substituted phenylene substituted in position 5 with SO_3Me;
Me is Li, K, Mg/2, Ca/2, Al/3, ammonium, mono-, di-, tri-, or tetraalkylammonium wherein the alkyl groups are (C_1-C_{22}) alkyl or (C_2-C_{10}) hydroxyalkyl, or mixtures thereof;
R^1, R^2, R^3, R^4, R^5 and R^6 are independently selected from H or (Ci-Cis) n- or iso-alkyl preferably methyl; and
R^7 is a linear or branched (C_1-C_{18}) alkyl, or a linear or branched (C_2-C_{30}) alkenyl, or a cycloalkyl group with 5 to 9 carbon atoms, a (C_6-C_{30}) aryl group or a (C_6-C_{50}) arylalkyl group preferably phenyl or benzyl;

preferably, wherein :

R^1 to R^6 independently are H or methyl,
R^7 is methyl,
a, b and c are a number from 1 to 20, preferably a and b are 1 and c is a number from 2 to 10,
d is a number between 1 and 25, preferably between 1 and 10, more preferably between 1 and 5,
e is a number between 1 and 30, preferably between 2 and 15, more preferably between 3 and 10, and
f is a number between 0.05 and 15, preferably between 0.1 and 10, more preferably between 0.25 and 3.

6. The method according to any preceding claims wherein the polymer premix comprises between 10% and 35%, preferably between 15% and 25%by weight of the polymer premix of the polyester terephthalate.

7. The method according to any preceding claims wherein the solvent comprises water, non-aqueous solvent, or a

mixture thereof, wherein the non-aqueous solvent preferably is selected from the group of ethanol, propanol preferably 1-propanol, butanol preferably 1-butanol, ethylene glycol, propylene glycol preferably 1,2-propylene glycol, dipropylene glycol, tripropylene glycol, polyethylene glycol, polypropylene glycol, glycerol, trimethylene glycol, or a mixture thereof, wherein preferably, the polyethylene glycol, polypropylene glycol or mixture thereof has an average molecular weight between 100 and 800 more preferably from 200 to 400 preferably wherein the non-aqueous solvent is selected from dipropylene glycol, tripropyleneglycol, propylene glycol preferably 1,2-propylene glycol, glycerol or a mixture thereof, most preferably a mixture of propylene glycol preferably 1,2-propylene glycol and glycerol.

8. The method according to claim 7, wherein the polymer premix comprises from 60% to 95% preferably from 65% to 90% more preferably from 75% to 85% by weight of the polymer premix of aqueous solvent, non-aqueous solvent or a mixture thereof, even more preferably the polymer premix comprises from 5% to 15% by weight of the polymer premix of water, from 15% to 25% by weight of the polymer premix of glycerol and from 60% to 80% by weight of the polymer premix of propylene glycol preferably 1,2-propylene glycol.

9. The method according to any preceding claims wherein in step c the polymer premix is added to the surfactant premix, preferably wherein the surfactant premix is mixed as the polymer premix is added.

10. The method according to any preceding claims wherein in step d, the polymer premix and the surfactant premix are mixed using a static mixer, a dynamic mixer or a mixture thereof.

11. The method according to any preceding claims wherein in step d, the polymer premix and the surfactant premix are mixed at a temperature of between 15°C and 30°C, more preferably between 17°C and 25°C.

12. The method according to any preceding claims wherein the non-soap anionic surfactant is selected from an alkyl benzene sulphonate, an alkyl sulphate, an alkoxylated alkyl sulphate or a mixture thereof.

13. The method according to any preceding claims wherein the surfactant premix comprises a solvent and wherein the solvent comprised in the surfactant premix may be selected from water, ethanol, propanol preferably 1-propanol, butanol preferably 1-butanol, ethylene glycol, propylene glycol preferably 1,2-propylene glycol, dipropylene glycol, polyethyleneglycol or polypropyleneglycol preferably with an average molecular weight between 100 and 800 preferably 200 and 400, glycerol, trimethylene glycol, or a mixture thereof, preferably water, propylene glycol preferably 1,2-propylene glycol, dipropylene glycol, tripropylene glycol, glycerol and a mixture thereof.

14. The method according to any preceding claims wherein the polymer premix is prepared at a temperature of above the glass transition temperature of the polyester terephthalate, preferably wherein the polymer premix is prepared at a temperature of between 50°C and 80°C, more preferably between 50°C and 70°C, even more preferably between 50°C and 65°C.

15. The method according to any preceding claims, wherein the liquid detergent composition is collected in a water-soluble unit dose article wherein the detergent composition is contained in an internal compartment enclosed by a water-soluble film.

Patentansprüche

1. Verfahren zum Herstellen einer undurchsichtigen flüssigen Waschmittelzusammensetzung, umfassend die Schritte;

a. Vorbereiten einer Polymervormischung, wobei die Polymervormischung umfasst

i. zwischen zu 5 Gew.-% und 40 Gew.-% der Polymervormischung ein anionisches Polyesterterephthalatpolymer, wobei das anionische Polyesterterephthalatpolymer ein Polyesterterephthalatgrundgerüst aufweist, das mit einer oder mehreren anionischen Gruppen gepfropft ist;
ii. zwischen zu 60 Gew.-% und 95 Gew.-% der Polymervormischung ein Lösungsmittel, wobei das Lösungsmittel ein wässriges Lösungsmittel, ein nichtwässriges Lösungsmittel oder eine Mischung davon ist;

b. Vorbereiten einer Tensidvormischung, wobei die Tensidvormischung zwischen zu 10 Gew.-% und 70 Gew.-%, vorzugsweise zwischen zu 20 Gew.-% und 65 Gew.-%, mehr bevorzugt zwischen zu 40 Gew.-% und 60 Gew.-% der Tensidvormischung ein Nicht-Seifen-Tensid umfasst, das Nicht-Seifen-Tensid vorzugsweise um-

fassend zwischen zu 50 Gew.-% und 100 Gew.-%, bevorzugt zwischen zu 70 Gew.-% und 100 Gew.-%, mehr bevorzugt zwischen zu 90 Gew.-% und 100 Gew.-% des Nicht-Seifen-Tensids ein anionischen Nicht-Seifen-Tensid, das anionische Nicht-Seifen-Tensid umfassend die protonierte Form des anionischen Nicht-Seifen-Tensids, die neutralisierte Salzform eines anionischen Nicht-Seifen-Tensids oder eine Mischung davon, vorzugsweise wobei die Vormischung des anionischen Nicht-Seifen-Tensids zwischen zu 50 Gew.-% und 90 Gew.-% des anionischen Nicht-Seifen-Tensids die neutralisierte Salzform eines anionischen Nicht-Seifen-Tensids umfasst;
c. Kombinieren der Polymervormischung und der Tensidvormischung in einem Gewichtsverhältnis der Polymervormischung zu der Tensidvormischung von 2 : 1 und 1 : 25, vorzugsweise zwischen 1,5 : 1 und 1 : 10;
d. Mischen der Polymervormischung und der Tensidvormischung;
e. optional Hinzufügen eines oder mehrerer Zusatzbestandteile;
f. Sammeln der endgültigen undurchsichtigen flüssigen Waschmittelzusammensetzung;
wobei mit "undurchsichtig" eine flüssige Zusammensetzung gemeint ist, die weniger als 1 % Durchlässigkeit aufweist, gemessen unter Verwendung eines ColorQuest XE-Spektralphotometers, unter Verwendung von 2,5 mL PS-Küvetten (1 cm Weglänge), einen Bereich von 400 bis 700 nm messend, wenn unverdünnt gemessen.

2. Verfahren nach Anspruch 1, wobei das Verfahren ein ununterbrochenes Verfahren oder ein Charge-Verfahren ist.

3. Verfahren nach einem der vorstehenden Ansprüche, wobei die Polymervormischung einen pH-Wert zwischen 4 und 8, vorzugsweise zwischen 5 und 7 aufweist.

4. Verfahren nach einem der vorstehenden Ansprüche, wobei die Tensidvormischung Monoethanolamin, Triethanolamin, ein Alkalimetall oder eine Mischung davon umfasst.

5. Verfahren nach einem der vorstehenden Ansprüche, wobei das Polyesterterephthalat die Struktureinheiten (I) bis (III) umfasst:

(I) $-[(OCHR1\text{-}CHR^2)_a\text{-}O\text{-}OC\text{-}Ar\text{-}CO\text{-}]_d$

(II) $-[(OCHR^3\text{-}CHR^4)_b\text{-}O\text{-}OC\text{-}sAr\text{-}CO\text{-}]_e$

(III) $-[(OCHR^5\text{-}CHR^6)_c\text{-}OR^7]_f$

wobei:

a, b und c von 1 bis 200 betragen;
d, e und f von 1 bis 50 betragen;
Ar ein 1,4-substituiertes Phenylen ist;
sAr 1 ,3-substituiertes Phenylen ist, das an Position 5 mit SOsMe substituiert ist;
Me für Li, K, Mg/2, Ca/2, Al/3, Ammonium, Mono-, Di-, Tri- oder Tetraalkylammonium steht, wobei die Alkylgruppen (C_1-C_{22})-Alkyl oder (C_2-C_{10})-Hydroxyalkyl oder Mischungen davon sind;
R^1, R^2, R^3, R^4, R^5 und R^6 unabhängig voneinander aus H oder (C_1-C_{18})n- oder Isoalkyl, vorzugsweise Methyl, ausgewählt sind; und R^7 ein lineares oder verzweigtes (C_1-C_{18})-Alkyl oder ein lineares oder verzweigtes (C_2-C_{30})-Alkenyl oder eine Cycloalkylgruppe mit 5 bis 9 Kohlenstoffatomen, eine (C_6-C_{30})-Arylgruppe oder eine (C_6-C_{50})-Arylalkylgruppe, vorzugsweise Phenyl oder Benzyl, ist;
vorzugsweise, wobei:

R^1 bis R^6 unabhängig H oder Methyl sind,
R^7 Methyl ist,
a, b und c eine Zahl von 1 bis 20 sind, vorzugsweise a und b 1 sind und c eine Zahl von 2 bis 10 ist,
d eine Zahl zwischen 1 und 25, vorzugsweise zwischen 1 und 10, mehr bevorzugt zwischen 1 und 5 ist,
e eine Zahl zwischen 1 und 30, vorzugsweise zwischen 2 und 15, mehr bevorzugt zwischen 3 und 10 ist und
f eine Zahl zwischen 0,05 und 15, vorzugsweise zwischen 0,1 und 10, mehr bevorzugt zwischen 0,25 und 3 ist.

6. Verfahren nach einem der vorstehenden Ansprüche, wobei die Polymervormischung zwischen zu 10 Gew.-% und 35 Gew.-%, vorzugsweise zwischen zu 15 Gew.-% und 25 Gew.-% der Polymervormischung das Polyesterterephtalat umfasst.

7. Verfahren nach einem der vorstehenden Ansprüche, wobei das Lösungsmittel Wasser, nicht wässriges Lösungsmittel oder eine Mischung davon umfasst, wobei das nichtwässrige Lösungsmittel vorzugsweise ausgewählt ist aus der Gruppe von Ethanol, Propanol, vorzugsweise 1-Propanol, Butanol, vorzugsweise 1-Butanol, Ethylenglycol, Propylenglycol, vorzugsweise 1,2-Propylenglycol, Dipropylenglycol, Tripropylenglycol, Polyethylenglycol, Polypropylenglycol, Glycerin, Trimethylenglycol oder einer Mischung davon, wobei vorzugsweise das Polyethylenglycol, das Polypropylenglycol oder die Mischung davon ein durchschnittliches Molekulargewicht zwischen 100 und 800, mehr bevorzugt von 200 bis 400 aufweist, vorzugsweise wobei das nichtwässrige Lösungsmittel ausgewählt ist aus Dipropylenglycol, Tripropylenglycol, Propylenglycol, vorzugsweise 1 ,2-Propylenglycol, Glycerin oder einer Mischung davon, am meisten bevorzugt einer Mischung aus Propylenglycol, vorzugsweise 1,2-Propylenglycol und Glycerin.

8. Verfahren nach Anspruch 7, wobei die Polymervormischung von zu 60 Gew.-% bis 95 Gew.-%, vorzugsweise von zu 65 Gew.-% bis 90 Gew.-%, mehr bevorzugt von zu 75 Gew.-% bis 85 Gew.-% der Polymervormischung wässriges Lösungsmittel, nicht wässriges Lösungsmittel oder eine Mischung davon umfasst, noch mehr bevorzugt die Polymervormischung von zu 5 Gew.-% bis 15 Gew.-% der Polymervormischung Wasser, von zu 15 Gew.-% bis 25 Gew.-% der Polymervormischung Glycerin und von zu 60 Gew.-% bis 80 Gew.-% der Polymervormischung Propylenglycol, vorzugsweise 1 ,2-Propylenglycol umfasst.

9. Verfahren nach einem der vorstehenden Ansprüche, wobei in Schritt c die Polymervormischung zu der Tensidvormischung hinzugefügt wird, vorzugsweise wobei die Tensidvormischung gemischt wird, wenn die Polymervormischung hinzugefügt wird.

10. Verfahren nach einem der vorstehenden Ansprüche, wobei in Schritt d die Polymervormischung und die Tensidvormischung unter Verwendung eines statischen Mischers, eines dynamischen Mischers oder einer Mischung davon gemischt werden.

11. Verfahren nach einem der vorstehenden Ansprüche, wobei in Schritt d die Polymervormischung und die Tensidvormischung bei einer Temperatur zwischen 15 °C und 30 °C, mehr bevorzugt zwischen 17°C und 25° C gemischt werden.

12. Verfahren nach einem der vorstehenden Ansprüche, wobei das anionische Nicht-Seifen-Tensid aus einem Alkylbenzolsulfonat, einem Alkylsulfat, einem alkoxylierten Alkylsulfat oder einer Mischung davon ausgewählt ist.

13. Verfahren nach einem der vorstehenden Ansprüche, wobei die Tensidvormischung ein Lösungsmittel umfasst und wobei das Lösungsmittel, das in der Tensidvormischung umfasst ist, aus Wasser, Ethanol, Propanol, vorzugsweise 1-Propanol, Butanol, vorzugsweise 1-Butanol, Ethylenglycol, Propylenglycol, vorzugsweise 1,2-Propylenglycol, Dipropylenglycol, Polyethylenglycol oder Polypropylenglycol, vorzugsweise mit einem mittleren Molekulargewicht zwischen 100 und 800, vorzugsweise 200 und 400, Glycerin, Trimethylenglycol oder einer Mischung davon, vorzugsweise Wasser, Propylenglycol, vorzugsweise 1,2-Propylenglycol, Dipropylenglycol, Tripropylenglycol, Glycerin und einer Mischung davon ausgewählt werden kann.

14. Verfahren nach einem der vorstehenden Ansprüche, wobei die Polymervormischung bei einer Temperatur von über der Glasübergangstemperatur des Polyesterterephthalats vorbereitet wird, vorzugsweise wobei die Polymervormischung bei einer Temperatur zwischen 50 °C und 80 °C, mehr bevorzugt zwischen 50 °C und 70 °C, noch mehr bevorzugt zwischen 50 °C und 65 °C vorbereitet wird.

15. Verfahren nach einem der vorstehenden Ansprüche, wobei die flüssige Waschmittelzusammensetzung in einem wasserlöslichen Einheitsdosisartikel gesammelt wird, wobei die Waschmittelzusammensetzung in einer inneren Kammer enthalten ist, die von einer wasserlöslichen Folie umschlossen ist.

Revendications

1. Procédé de fabrication d'une composition de détergent liquide opaque comprenant les étapes consistant à ;

   a. préparer un prémélange polymère, dans lequel le prémélange polymère comprend

      i. entre 5 % et 40 % en poids du prémélange polymère d'un polymère de téréphtalate de polyester anionique,

dans lequel le polymère de téréphtalate de polyester anionique a un squelette de téréphtalate de polyester greffé avec un ou plusieurs groupes anioniques ;
ii. entre 60 % et 95 % en poids du prémélange polymère d'un solvant, dans lequel le solvant est un solvant aqueux, un solvant non aqueux ou un mélange de ceux-ci ;

b. préparer un prémélange de tensioactif, dans lequel le prémélange de tensioactif comprend entre 10 % et 70 %, de préférence entre 20 % et 65 %, plus préférablement entre 40 % et 60 % en poids du prémélange de tensioactif d'un tensioactif non savonneux, le tensioactif non savonneux comprenant de préférence entre 50 % et 100 %, plus préférablement entre 70 % et 100 %, encore plus préférablement entre 90 % et 100 % en poids du tensioactif non savonneux d'un tensioactif anionique non savonneux, le tensioactif anionique non savonneux comprenant la forme protonée du tensioactif anionique non savonneux, la forme de sel neutralisée d'un tensioactif anionique non savonneux ou un mélange de ceux-ci, de préférence dans lequel le prémélange de tensioactif anionique non savonneux comprend entre 50 % et 90 % en poids du tensioactif anionique non savonneux de la forme de sel neutralisée d'un tensioactif anionique non savonneux ;
c. combiner le prémélange polymère et du prémélange tensioactif dans un rapport de poids du prémélange polymère au prémélange tensioactif de 2:1 et 1:25, de préférence entre 1,5:1 et 1:10 ;
d. mélanger le prémélange polymère et du prémélange de tensioactif ;
e. facultativement ajouter un ou plusieurs ingrédients additifs ;
f. collecter la composition de détergent liquide opaque finale ;
dans lequel, par « opaque », on entend une composition liquide ayant moins de 1 % de transmittance mesurée à l'aide d'un spectrophotomètre ColorQuest XE, en utilisant des cuvettes de 2,5 mL PS (longueur de trajet de 1 cm), mesurant une plage allant de 400 à 700 nm lorsqu'elle est mesurée pure.

2. Procédé selon la revendication 1, dans lequel le procédé est un procédé continu ou un procédé discontinu.

3. Procédé selon l'une quelconque des revendications précédentes dans lequel le prémélange polymère a un pH compris entre 4 et 8, de préférence entre 5 et 7.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel le prémélange de tensioactif comprend de la monoéthanolamine, de la triéthanolamine, un métal alcalin ou un mélange de ceux-ci.

5. Procédés selon l'une quelconque des revendications précédentes dans lesquels le téréphtalate de polyester comprend les unités structurelles suivantes (I) à (III) :

(I) $-[(OCHR1\text{-}CHR^2)_a\text{-}O\text{-}OC\text{-}Ar\text{-}CO\text{-}]_d$

(II) $-[(OCHR^3\text{-}CHR^4)_b\text{-}O\text{-}OC\text{-}sAr\text{-}CO\text{-}]_e$

(III) $-[(OCHR^5\text{-}CHR^6)_c\text{-}OR^7]_f$

où :

a, b et c vont de 1 à 200 ;
d, e et f vont de 1 à 50 ;
Ar est un phénylène à substitution 1,4 ;
sAr est un phénylène à substitution 1,3 substitué en position 5 par SO_3Me ;
Me est Li, K, Mg/2, Ca/2, Al/3, ammonium, mono-, di-, tri-, ou tétra-alkylammonium où les groupes alkyle sont un alkyle en (C_1 à C_{22}) ou un hydroxyalkyle en (C_2 à C_{10}), ou leurs mélanges ;
R^1, R^2, R^3, R^4, R^5 et R^6 sont indépendamment sélectionnés parmi H ou un iso-alkyle en (C_1 à C_{18}) n-, de préférence méthyle ; et R^7 est un alkyle linéaire ou ramifié en (C_1 à C_{18}), ou un alcényle linéaire ou ramifié en (C_2 à C_{30}), ou un groupe cycloalkyle avec 5 à 9 atomes de carbone, un groupe aryle en (C_6 à C_{30}) ou un groupe arylalkyle en (C_6 à C_{50}) de préférence phényle ou benzyle.
de préférence, où :

R^1 à R^6 sont indépendamment H ou méthyle,
R^7 est méthyle,
a, b et c sont un nombre de 1 à 20, de préférence a et b sont 1 et c est un nombre de 2 à 10,
d est un nombre entre 1 et 25, de préférence entre 1 et 10, plus préférablement entre 1 et 5,

e est un nombre entre 1 et 30, de préférence entre 2 et 15, plus préférablement entre 3 et 10, et
f est un nombre entre 0,05 et 15, de préférence entre 0,1 et 10, plus préférablement entre 0,25 et 3.

6. Procédé selon l'une quelconque des revendications précédentes dans lequel le prémélange polymère comprend entre 10 % et 35 %, de préférence entre 15 % et 25 % en poids du prémélange polymère du téréphtalate de polyester.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel le solvant comprend de l'eau, un solvant non aqueux, ou un mélange de ceux-ci, dans lequel le solvant non aqueux est de préférence sélectionné parmi le groupe constitué par l'éthanol, le propanol, de préférence le 1-propanol, le butanol, de préférence le 1-butanol, l'éthylène glycol, le propylène glycol, de préférence le 1,2-propylène glycol, le dipropylène glycol, le tripropylène glycol, le polyéthylène glycol, le polypropylène glycol, le glycérol, le triméthylène glycol, ou un mélange de ceux-ci, dans lequel de préférence, le polyéthylène glycol, le polypropylène glycol ou un mélange de ceux-ci a un poids moléculaire moyen entre 100 et 800 plus préférablement de 200 à 400, de préférence dans lequel le solvant non aqueux est sélectionné parmi le dipropylène glycol, le tripropylène glycol, le propylène glycol de préférence le 1,2-propylène glycol, le glycérol ou un mélange de ceux-ci, le plus préférablement un mélange de propylène glycol de préférence de 1,2-propylène glycol et de glycérol.

8. Procédé selon la revendication 7, dans lequel le prémélange polymère comprend de 60 % à 95 % de préférence de 65 % à 90 % plus préférablement de 75 % à 85 % en poids du prémélange polymère de solvant aqueux, de solvant non aqueux ou d'un mélange de ceux-ci, encore plus préférablement le prémélange polymère comprend de 5 % à 15 % en poids du prémélange polymère d'eau, de 15 % à 25 % en poids du prémélange polymère de glycérol et de 60 % à 80 % en poids du prémélange polymère de propylène glycol de préférence de 1,2-propylène glycol.

9. Procédé selon l'une quelconque des revendications précédentes dans lequel à l'étape c le prémélange polymère est ajouté au prémélange de tensioactif, de préférence dans lequel le prémélange de tensioactif est mélangé au fur et à mesure que le prémélange polymère est ajouté.

10. Procédé selon l'une quelconque des revendications précédentes, dans lequel à l'étape d, le prémélange polymère et le prémélange de tensioactif sont mélangés à l'aide d'un mélangeur statique, d'un mélangeur dynamique ou d'un mélange de ceux-ci.

11. Procédé selon l'une quelconque des revendications précédentes, dans lequel à l'étape d, le prémélange polymère et le prémélange de tensioactif sont mélangés à une température comprise entre 15 °C et 30 °C, plus préférablement entre 17 °C et 25 ° C.

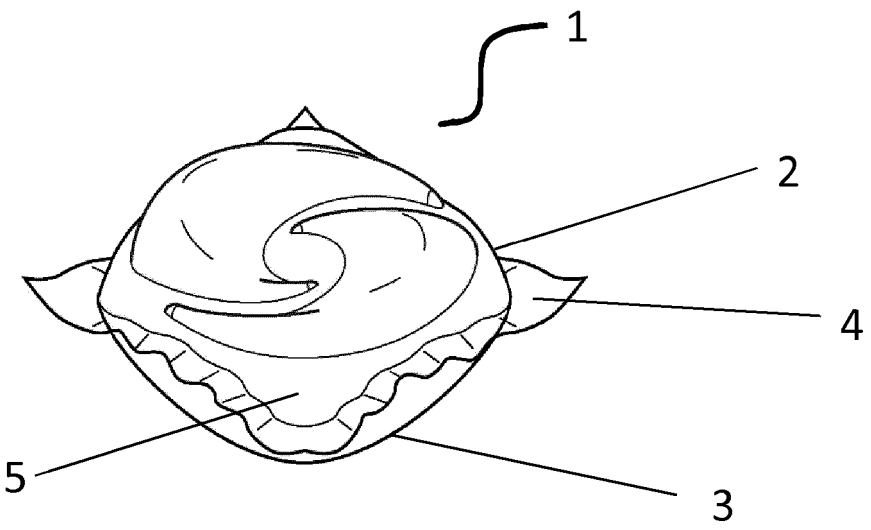
12. Procédé selon l'une quelconque des revendications précédentes, dans lequel le tensioactif anionique non savonneux est sélectionné parmi un sulfonate d'alkylbenzène, un sulfate d'alkyle, un sulfate d'alkyle alcoxylé ou un mélange de ceux-ci.

13. Procédé selon l'une quelconque des revendications précédentes dans lequel le prémélange de tensioactif comprend un solvant et dans lequel le solvant compris dans le prémélange de tensioactif peut être sélectionné parmi l'eau, l'éthanol, le propanol, de préférence le 1-propanol, le butanol, de préférence le 1-butanol, l'éthylène glycol, le propylène glycol, de préférence le 1,2-propylène glycol, le dipropylène glycol, le polyéthylène glycol ou le polypropylène glycol, de préférence avec un poids moléculaire moyen entre 100 et 800, de préférence 200 et 400, le glycérol, le triméthylène glycol, ou un mélange de ceux-ci, de préférence de l'eau, du propylène glycol, de préférence du 1,2-propylène glycol, du dipropylène glycol, du tripropylène glycol, du glycérol et un mélange de ceux-ci.

14. Procédé selon l'une quelconque des revendications précédentes dans lequel le prémélange polymère est préparé à une température supérieure à la température de transition vitreuse du téréphtalate de polyester, de préférence dans lequel le prémélange polymère est préparé à une température comprise entre 50 °C et 80 °C, plus préférablement entre 50 °C et 70 °C, encore plus préférablement entre 50 °C et 65 ° C.

15. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition de détergent liquide est recueillie dans un article en dose unitaire soluble dans l'eau dans lequel la composition de détergent est contenue dans un compartiment interne entouré par un film soluble dans l'eau.

FIG. 1



REFERENCES CITED IN THE DESCRIPTION

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