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(54) Title: SURFACTANT COMPOSITIONS COMPRISING CURVED LAMELLAR ELEMENTS AS A VISUAL CUE

(57) Abstract: A packaged, pourable liquid or gel, surfactant-containing formulation comprising a visual cue, wherein the visual cue comprises a plurality of sheet-like elements of dispersed material, preferably formed from modified poly vinyl alcohol, having opposed surfaces wherein the surfaces of the elements are configured such that when an element is located adjacent with a flat inner surface of the package, the element can only contact the inner surface of the packaging over less than 50% of a surface of the element.

SURFACTANT COMPOSITIONS COMPRISING CURVED LAMELLAR ELEMENTS AS A VISUAL CUE

FIELD OF INVENTION

The present invention relates to pourable surfactant compositions comprising lamellar elements as a visual cue, in particular liquid laundry detergent compositions (including both powders and liquids), processes for making the elements which comprise the visual cue, the elements per-se, and to the use of said elements to deliver a benefit agent to fabric or to a wash and/or rinse medium during a wash process.

BACKGROUND

Pourable, laundry treatment compositions (particulate or liquid) generally contain ingredients which provide a benefit to laundered clothes. Examples of such ingredients include, but are not limited to, perfumes, enzymes, bleaches, shading pigments and dyes and fabric conditioning agents. These materials are also often the more expensive components of the laundry composition and are known as "benefit agents". It can be advantageous to protect (for example by encapsulation) benefit agents when they are included in formulations due to potential incompatibility with other ingredients in the formulation. This incompatibility can cause the formulation to be unstable, or involve unwanted reactivity of the benefit agent with other formulation components and so cause the efficacy of the benefit agent, or other product properties, to be adversely effected.

It has been proposed to incorporate these benefit agents in a so-called "visual cue", and/or, where the benefit agent is stable in the composition, to include in the

formulation visual cues to indicate that the benefit agent is present. The elements comprising the cue are delivered as part of the product dose and are soluble at some stage of the wash. One such visual cue is the coloured "speckle" which is often incorporated in enzyme containing laundry powders. This is often a particle of a salt such as sodium carbonate coloured with a suitable dye and does not itself contain the enzyme.

US2003/1 441 6 1A discloses particulate laundry detergent compositions especially high bulk density powders and tablets which contain low levels of aloe vera or another herbal extract beneficial to the skin. The herbal extract is preferably present in the form of coloured (preferably green) speckles.

Other visual cues that have been proposed or used have included beads, lamellar shapes such as flat rings and various "flower" shapes.

GB2358403A discloses a particulate laundry detergent composition which comprises a major proportion of white or light-coloured particles and a minor proportion of visually contrasting bodies of significantly larger average particle size in at least one dimension than the average particle size of the white or light-coloured particles. Preferably the bodies are of regular shape and uniform size and formed of brightly-coloured material which is preferably highly reflective and/or fluorescent. The visually contrasting bodies provide strong cues to the consumer for example to demonstrate the presence of a beneficial ingredient even if present in very small amounts.

GB2358404A discloses a particulate laundry detergent composition which comprises a major proportion of white or light-coloured particles and a minor proportion of visually contrasting particles of material capable of imparting a colour to the resulting solution when the composition is dissolved in water. The visually contrasting particles may contain a fluorescent material yielding fluorescent wash

liquor or may contain a pH indicator which gives a coloured solution at high pH but a colourless or differently coloured solution at lower pH values. The visually contrasting particles and the visual effects in the wash liquor provide cues to the consumer for example to demonstrate the presence of a beneficial ingredient or to indicate that more product should be added.

WO2009/0471 25A discloses a granular laundry detergent composition comprising at least 5 wt percent surfactant optional builder optional further cleaning ingredients a perfume and 0.01 to 10 wt percent of visual cues wherein the visual cue is shaped like a flower with petals, is coloured to have a naturally occurring petal colour and wherein the perfume is floral in essence. "Lamellar Visual Cue" is defined in the document as meaning visual cue particles in the form of *planar* film and flower shaped (or flower like) is defined as meaning a *planar* film shape that has the outline of the shape of a flower with petals. There is no suggestion that the flowers are other than flat, planar shapes.

WO2009/0471 26A discloses granular detergent composition comprising 0.1 to 10 wt percent soluble coloured lamellar visual cues made from a soluble film each soluble coloured lamellar visual cue having a *planar* cross-sectional area of from 5mm2 to 100 mm2 the relative density of the film being from 0.2 to 0.8 kg/l and the film comprising 10 to 90 wt percent surfactant.

Known lamellar elements for use as a cue are typically manufactured by cutting a shape out of a flat sheet of a suitable material.

WO2009/0471 24A discloses a laundry treatment composition comprising a laundry treatment base and from 0.01 to 10 wt percent of contrastingly coloured concave lamellar film particles which have a planar surface and a planar periphery and wherein: a) the periphery is shaped so that at least one straight line drawn through the planar surface intersects the periphery at more than two places; b) the

concave lamellar film particles may be tiled; c) the concave lamellar film particles contrast in colour with the laundry treatment base whereby the concave lamellar film particles function as a visual cue. In this citation "concave" refers to the edge of the cue.

WO2006/07941 6 discloses a laundry detergent composition with lamellar body additives intended especially for use in powder formulations. The specification is concerned with the problem of the additives sticking in the drawer of a washing machine when the powder is dispensed. This problem is solved by making highly soluble cues using gum arabic. It is suggested that the adhesion of additives to the drawer during powder dispensing can further be reduced by making the cues curved with a radius of 0.5-2.0 meters, but the specification contain no teaching as to how such curved cues should be made.

The elements making up the known cues have suffered from the disadvantage, particularly in liquid compositions, that they tend to stick to the inside of the packaging and to each other, so that dosage of the individual elements is irregular. Where the cue contains a benefit agent, this leads to a variable level of benefit agent being delivered, possible wastage when the package is "empty", and creates a negative impression in the mind of the user, reducing the probability that the product will be purchased again. Making cues of high solubility will not directly solve this problem as the cues would dissolve in the product.

SUMMARY OF INVENTION

We have determined that the adhesion of lamellar elements forming a water-soluble visual cue to packaging surfaces and to each other can be reduced by using elements which are not "flat".

Accordingly, the present invention provides a packaged surfactant-containing, pourable liquid or gel, formulation comprising a visual cue, wherein the visual cue comprises a plurality of sheet-like elements of dispersed material, having opposed surfaces wherein the surfaces of the elements are configured such that when an element is located adjacent with a flat inner surface of the package, the elements can only contact the inner surface of the packaging over less than 50% of a surface of the element.

A benefit of the non-flat elements is that they not only show less of tendency to stick to the inner surface of the packaging but also less of a tendency to clump together. This improves the visual aspects of the cue and assists in ensuring that even dosage of the elements is maintained. Even dosage of cue elements is a particularly important feature of the product for end-users. The "flat inner surface of the packaging" referred to herein be notional as the packaging may not have a surface which is perfectly flat. The relevant test is to place the cue against a flat surface of the packaging material.

"Pourable liquid" as defined herein refers to a liquid having a viscosity of less than about 2000 mPa.s at 25°C and a shear rate of 20 sec-1 . In some embodiments, the viscosity of the pourable liquid may be in the range of from about 200 to about 1000 mPa.s at 25°C at a shear rate of 20 sec-1 . In some embodiments, the viscosity of the pourable liquid may be in the range of from about 200 to about 500 mPa.s at 25°C at a shear rate of 20 sec-1 . "Gel" as defined herein refers to a transparent or translucent liquid having a viscosity of greater than about 2000 mPa.s at 25°C and at a shear rate of 20 sec-1 . In some embodiments, the viscosity of the gel may be in the range of from about 3000 to about 10,000 mPa.s at 25°C at a shear rate of 20 sec-1 and greater than about 5000 mPa.s at 25°C at a shear rate of 0.1 sec-1 . Although the cue can also be used in powders, pourable liquid formulations are preferred. Pourable detergent liquids generally have a viscosity at 20 sec^{-1} of from 100 mPa.s to 2000 mPa.s.

Typically, a low viscosity liquid will contain a structuring agent to assist in keeping the cues in suspension. One type of structuring agent which is especially useful in the compositions of the present invention comprises non-polymeric (except for conventional alkoxylation), crystalline hydroxy-functional materials which can form thread-like structuring systems throughout the liquid matrix when they are crystallized within the matrix in situ. Such materials can be generally characterized as crystalline, hydroxyl-containing fatty acids, fatty esters or fatty waxes. Specific examples of preferred crystalline, hydroxyl-containing structurants include castor oil and its derivatives. Examples include mixtures of hydrogenated castor oil and its hydrolysis products, e.g. hydroxyl-stearic acid. Especially preferred are hydrogenated castor oil derivatives such as hydrogenated castor oil and hydrogenated castor wax. Commercially available, castor oil-based, crystalline, hydroxyl-containing structurants include THIXCIN® from Rheox, Inc. (now Elementis).

Preferably, the packaging is at least in part transparent. "Transparent" as used herein means that a composition, or a package according to the invention preferably has a transmittance of more than 25%, more preferably more than 30%, most preferably more than 40%, optimally more than 50% in the visible part of the spectrum (approx. 410-800 nm). Packaging materials with which this invention may be used include, but are not limited to: polypropylene (PP), polyethylene (PE), polycarbonate (PC), polyamides (PA) and/or polyethylene terephthalate (PETE), polyvinylchloride (PVC); and polystyrene (PS) or multilayer combinations.

The packaging may initially contain several doses of the surfactant composition. The invention is also applicable to so-called "unit-dose" liquid detergent compositions which comprise a water-soluble pouch containing a single dose of the surfactant composition, but the preferred format of the composition is one delivered as a plurality of doses of a liquid in a bottle or other such container.

Preferably the liquid is transparent. By transparent we mean that compositions of the invention are such that a sample of Arial 12 point printing can be read through a 1 centimeter cuvette containing the product (in the absence of the cue). A lamellar cue in a transparent liquid and an at least partly transparent container, provided that the elements do not cluster or adhere to the packaging, has a higher visual impact as compared to a cue comprising spheres consisting of the same volume of material.

A particularly preferred embodiment of the invention therefore provides a rigid, at least partially transparent container, which contains a transparent liquid laundry detergent composition, said composition having dispersed therein a plurality of sheet-like elements which are soluble in demineralised water but insoluble in the composition.

A further advantage that the cue provides is that the elements can be used to carry useful benefit agents (in particular dyes) which, if not protected from the composition, would significantly change its visual appearance.

Preferably the ratio of the average diameter of the elements (measured flat) to the thickness of the elements is greater than 5:1 , preferably greater than 8:1 .

Typically, the average diameter of the elements will be in the range 1-10mm, preferably 3-6mm. Elements smaller than 1mm have a low visual impact whereas ones larger than 10mm can lead to irregular dosing. Typically, the average thickness of the elements will be in the range 50-2000 microns, preferably 100-500 microns, more preferably 100-250 microns. As the elements get thicker they become more expensive in formulation terms and less easy to suspend. As the elements become thinner they have less benefit agent carrying capacity and more may be needed to deliver an appropriate level of benefit agent.

Appropriate curvature can be achieved by several geometries. Generally, the lamellar elements may be folded or curled. Where the elements are "star" or "flower"-shaped, the "petals" may curve in opposing directions. In the context of the present invention a star or flower shape is one which has a varying radius preferably such that it has rotational symmetry.

Processes for making the elements include both forming elements which are already curved prior to admixture with other formulation components and, in the alternative, forming elements which curve on exposure to one or more other components of the formulation.

A preferred step for forming non-flat elements is to dry them unevenly, such that regions on opposing faces of the element are dried at different rates. This causes the elements to curl slightly, either directly on drying or when exposed to the formulation.

Depending on the benefit agent being provided, the cue may be designed to dissolve in the early stages of the wash, or may be carried over into the rinse and subsequently dissolve in the rinse. Thus, a further advantage of the cue is that they can deliver benefit agents which are otherwise incompatible with a formulation's other components. For example, a cue can be used to deliver perfumes into the rinse and reduce the extent to which the perfumes are removed by the surfactants present in the main wash.

A preferred surfactant composition is a liquid laundry detergent composition comprising an anionic surfactant, a nonionic surfactant, or a mixture thereof.

It is particularly preferred that, for the embodiments as liquids laundry detergent compositions, the dosage in which the composition is applied is such that the wash liquor produced has a surfactant (other than soap) concentration of less

than 1 g/L, more preferably less than 0.5 g/L. This is a low level of surfactant as compared with conventional wash liquors. It is also preferred that the dosage of a liquid detergent product containing the cue is less than 30ml and preferably less than 25ml. This is a low dosage as compared with conventional doses of laundry compositions. Such low dosage and/or low surfactant products have environmental advantages in that the products require transport of smaller quantities of material and have a reduced usage of surfactants. However in order to persuade the user that the low dosage of product is effective, it is highly advantageous to include a visual cue comprising lamellar elements as this has a significant visual impact even in a low dosage of product.

DETAILED DESCRIPTION OF THE INVENTION

All amounts quoted are wt.% of total composition unless otherwise stated.

Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts or ratios of material or conditions of reaction, physical properties of materials and/or use are to be understood as modified by the word "about".

Elements forming the Cue:

Preferred elements are made from a material having a polymer backbone which is water soluble prior to being hydrophobically-modified.

By 'water-soluble' used herein in relation to the polymer, it is meant that the polymer should dissolve in water such that when 0.1 g, preferably 0.3g, more preferably 0.5g of polymer is placed into 1L of demineralised water at room temperature and shaken at 100 RPM on a rotator shaker at 20 Celsius for 2

hours, then removed from solution by filtering through a sieve or filter paper of appropriate size and dried, then the weight of the polymer removed is less than 95% by weight of that added. "Water-soluble", in the context of the cue means that the cue dissolves in the course of the wash.

By 'insoluble' used herein in relation to the modified polymer, it is meant that the polymer (and hence the elements of the cue formed from it) should not dissolve in surfactant solutions. Suitable polymers are ones which meet the condition that when 1g/L of the modified polymer is placed into an aqueous surfactant mixture (a mixture of linear alkylbenze sulfonate (LAS) and nonionic surfactant (which is a reaction product of aliphatic C12 to C15 primary linear alcohols with ethylene oxide (7 EO)) where the surfactant concentration is greater than 5g/l at room temperature and shaken at 100 RPM on a rotator shaker at 293K for 2 hours, then removed from solution by filtering through a sieve or filter paper of appropriate size and dried, then the weight of the modified polymer removed is within 95% by weight of that added.

Preferably, the cue is insoluble in an aqueous surfactant mixture (a mixture of linear alkylbenze sulfonate (LAS) and nonionic surfactant (which is a reaction product of aliphatic C12 to C15 primary linear alcohols with ethylene oxide (7 EO)) where the surfactant concentration is from 5 to 800g/l, more preferably greater from 5 to 500g/l, for example 50 to 500g/l.

Lower critical solution temperature (LCST) is a characteristic of a material that demonstrates good solubility in aqueous solutions at low temperatures, but separates from solution when the temperature is raised above the LCST (see Feil et al., Macromolecules 1993, *26,* 2496-2500). The 'aqueous solutions' where the LCST effect referred to is shown for polymers of the present invention include water and aqueous surfactant solutions (including aqueous surfactant mixtures). Preferred LCST ranges of the modified polymer are from 5 to 55°C, more

preferably from 5 to 50˚C. In a preferred embodiment of the invention, the modified polymer has a LCST in water of greater than 20˚C, more preferably greater than 30˚C.

Typically, the elements comprise a matrix of, for example, the polymer with multiple discrete benefit agent entities embedded in the matrix. The discrete benefit agent entities embedded in the matrix can be the same or different benefit agents. A discrete benefit entity means, for example, a particle comprising encapsulated perfume, or a droplet of softening oil.

The elements preferably comprises a matrix material, preferably a hydrophobically modified polymer (i), and a fabric benefit agent (ii), in a ratio of from 1:50 to 99:1 parts by weight, more preferably from 1:40 to 95:1 parts by weight.

Polymers suitable for use as whole or part of the backbone of the hydrophobically modified polymer are preferably selected from the group consisting of polyvinyl alcohol, polyvinyl acetate, cellulose ethers, polyethylene oxide, starch, polyvinylpyrrolidone, polyacrylamide, polyvinyl methyl ether-maleic anhydride, polymaleic anhydride, styrene maleic anhydride, hydroxyethylcellulose, methylcellulose, polyethylene glycols, carboxymethylcellulose, polyacrylic acid salts, alginates, acrylamide copolymers, guar gum, casein, ethylene-maleic anhydride resin series, polyethyleneimine, ethyl hydroxyethylcellulose, ethyl methylcellulose and hydroxyethyl methylcellulose. Copolymeric mixtures of polymers derived from the aforementioned backbones are also suitable. Preferably the polymer has a backbone and side chains comprising hydroxyl groups.

The most preferred backbone for the polymer comprises polyvinyl alcohol, and the polymer preferably has an average molecular weight of from 1,000 to 300,000 Daltons, preferably from 2,000 to 100,000 Daltons.

Polyvinyl alcohol (PVOH) can be supplied in a form comprising a certain amount of polyvinyl acetate (PVAc), in that a level of the hydroxyl groups (OH) of the PVOH material is substituted with acetate groups ($OCOCH_3$). Hydrolysis of PVAc is a common way to make PVOH. Thus the PVOH used herein generally comprise at some PVAc. The PVOH materials (either before or after hydrophobic modification) may comprise from 0.01 to 40% PVAc, preferably from 0.01 to 20%, more preferably from 0.1 to 15%, most preferably 0.5 to 10%, based on the % of the total number of monomers making up the polymer. As used herein, the term PVOH includes PVOH compounds with a PVAc level as previously defined. Particularly preferred materials have a "degree of hydrolysis" of from 85 to 99%.

The preferred polymer is modified to comprise hydrophobic substituents. One method of modification is detailed in example 1a.

Preferred hydrophobic derivatisation groups include those based on parent groups selected from acetals, ketals, esters, fluorinated organic compounds, ethers, alkanes, alkenes and aromatics.

Highly preferred hydrophobic substituents are hydrocarbyl groups of C_4 to C22 carbon chain length. These hydrocarbyl groups may be alkyl or alkenyl based, which can be straight chain, branched or comprise rings; it may also or alternatively incorporate aromatic moieties.

More preferably the hydrocarbyl group has a carbon chain length of from C_4 to C20, even more preferably from C_4 to C15, most preferably from C_4 to C10, for example, from C_4 to Cs.

Hydrocarbyl chain lengths greater than C22 are undesirable as the parent material from which the derivatising group is obtained reacts poorly or not at all with the

polymeric backbone. Hydrocarbyl groups shorter than C_4 provide negligible additional hydrophobicity.

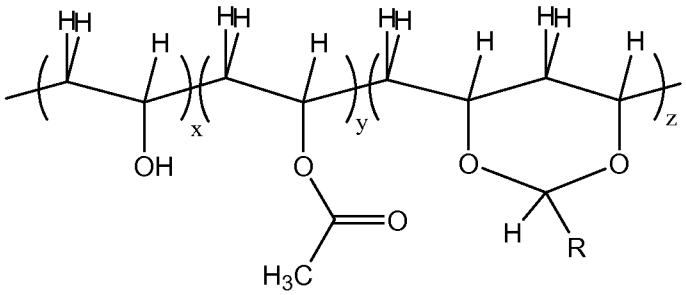
Especially preferred materials suitable for use to introduce the hydrophobic derivatisation groups onto the polymer are aldehydes such as butyraldehyde, octyl aldehyde, dodecyl aldehyde, 2-ethyl hexanal, cyclohexane carboxy-aldehyde, citral and 4-aminobutyraldehyde dimethyl acetal.

The hydrophobic material is preferably present in the polymer at a level from 0.1 to 40% by weight, based on the total weight of the polymer, more preferably from 2 to 30%, most preferably from 4 to 15%. In practice, with PVOH materials of relatively high degrees of hydrolysis, this means that preferred materials will have 5-15% of the -OH groups of the polymer replaced with the hydrophobe.

Where the polymeric backbone is based on polyvinyl alcohol (PVOH), the hydrophobic derivatisation material is preferably present at a level such that the number ratio of the hydrophobic groups to the free hydroxyl pairs on the backbone is from 1:3 to 1:30, more preferably from 1:4 to 1:20, most preferably 1:7 to 1:15.

Additional modifying groups may be present on the polymer backbone. For instance, amines may be preferably included as a modifying group since this makes the polymer more soluble in response to, for instance, the change in pH and/or ionic strength from the wash to the rinse liquor.

A particularly preferred polymer particle comprises a hydrophobic modified polymer of formula:



wherein the average number ratio of z to x is within the range of from 1:200 to 1:6, y is in the range of from 0.01 to 20% based on the % of total number of monomers making up the polymer (x + y + z), and R is a hydrophobic group being an alkyl or alkenyl group having from 3 to 21 carbon atoms, preferably from 3 to 6 carbon atoms. Most preferably R is C_3H_7.

Benefit Agents

While benefit agents are an optional feature of the elements comprising the cue, their presence is highly preferred in at least some of the elements. Various benefit agents can be incorporated into the elements. Any compatible benefit agent which can provide a benefit to a substrate which is treated with a surfactant composition can be used. Preferred benefit agents are in the laundry field, for example fabric benefit agents, and benefit agents which provide a benefit to a laundry wash and/or rinse medium.

Preferred examples include perfumes (both free and encapsulated), enzymes, antifoams, shading dyes and/or pigments, detergency builders, fabric conditioning agents (for example water-insoluble quaternary ammonium materials and/or silicones), sunscreens, antioxidants, reducing agents, sequestrants, colour care additives, density matching polymers, photobleaches, unsaturated oils, emollients and antimicrobial agents.

A preferred embodiment of the invention includes a disintegrant in the elements in addition to the benefit agent.

The elements are particularly suitable for use with particulate benefit agents (for example encapsulated perfumes) or water soluble/dispersible benefit agents.

Plasticiser and/or Crystallinity Disruptor

Where the elements of the cue are formed from a polymer the polymer preferably incorporates a plasticiser and/or crystallinity disruptor.

It is to be understood that the term "plasticiser" and phrase "crystallinity disruptor" are interchangeable such that a reference to one is an implicit reference to the other.

The plasticiser influences the way the polymer chains react to external factors such as compression and extensional forces, temperature and mechanical shock by controlling the way that the chains distort/realign as a consequence of there intrusions and their propensity to recover to their former state. The key feature of plasticisers is that they are highly compatible with the polymer cue and are normally hydrophilic in nature. The plasticiser will depend on the nature of the polymer making up the elements of the cue.

Generally, plasticisers suitable for use with PVOH-based polymer cues have -OH groups in common with the $-CH_2-CH(OH)-CH_2-CH(OH)-$ polymer chain of the polymer particle.

Their mode of functionality is to introduce short chain hydrogen bonding within the hydroxyl groups and this weakens adjacent chain interactions which inhibits swelling of the aggregate polymer mass - the first stage of polymer dissolution.

Water itself is a suitable plasticiser for PVOH polymer particles but other common plasticisers include: polyhydroxy compounds e.g. glycerol, trimethylolpropane, diethylene glycol, triethylene glycol, sorbitol, dipropylene glycol, polyethylene glycol, starches e.g. starch ether, esterified starch, oxidized starch and starches from potato, tapioca and wheat, cellulosics/carbohydrates e.g. amylopectin, dextrin, carboxymethylcellulose and pectin. Amines are particularly preferred plasticisers.

SURFACTANT COMPOSITIONS

Various preferred surfactants are listed below. Particularly preferred surfactant compositions are those in the field of home and personal care, especially in the field of laundry, hair and oral care. The fully formulated compositions preferably comprise from 0.05 to 10 wt.% of the elements comprising the cue as previously described, and from 2 to 70 wt.%, more preferably 10 to 30 wt.% of a surfactant selected from an anionic surfactant, a nonionic surfactant, a cationic surfactant, or a mixture thereof. The elements forming the cue are more preferably present in the surfactant composition at a level of from 0.2 to 10 wt.%, most preferably from 0.2 to 5 wt.%.

LAUNDRY TREATMENT COMPOSITIONS

The cue is preferably incorporated in a laundry treatment composition.

The laundry treatment composition may, for example, take the form of an isotropic liquid, a surfactant-structured liquid, a granular, spray-dried, dry-blended or other particulate material. The composition is preferably a liquid or gel laundry composition.

The laundry treatment composition may be a detergent composition for use in the main wash; alternatively it may be a composition for addition to a rinse cycle, for example a fabric conditioner.

PROCESS OF PREPARATION

In one embodiment, the invention provides a process for making the elements of the cues of the invention, the process comprising the steps of:

(a) provision of an aqueous mixture of a hydrophobically modified polymer as defined herein, and a fabric benefit agent;

(b) drying the aqueous mixture to remove whole or part of the water from the aqueous mixture to form a sheet;

(c) forming elements from the sheet in the desired shape and/or size; and,

(d) optionally, further drying the elements.

The curvature of the elements may be instilled during the forming step (c), in the alternative the elements may adopt a curved shape when exposed to other formulation components of the end product. A preferred embodiment of the invention provides a process for making the elements in which the drying step (b) or (d) is at least in part conducted unevenly, preferably with one face being dried more quickly than the other. This causes the elements to curl, either on drying or when exposed to other components of the end formulation.

SURFACTANT

In general, the nonionic and anionic surfactants of the surfactant system may be chosen from the surfactants described "Surface Active Agents" Vol. 1, by Schwartz & Perry, Interscience 1949, Vol. 2 by Schwartz, Perry & Berch, Interscience 1958, in the current edition of "McCutcheon's Emulsifiers and Detergents" published by Manufacturing Confectioners Company or in "Tenside-Taschenbuch", H. Stache, 2nd Edn., Carl Hauser Verlag, 1981.

Preferably the surfactants used are saturated.

Suitable nonionic detergent compounds which may be used include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide.

Non-limiting examples of nonionic detergent compounds include: **C12-C18** alkyl ethoxylates, such as, NEODOL® nonionic surfactants from Shell and LUTENSOL® XL and LUTENSOL® XP from BASF; C10-CI2 alkyl phenol alkoxylates wherein the alkoxylate units are a mixture of ethoxy and propoxy units; **C12-C18** alcohol and C6-C12 alkyl phenol condensates with ethylene oxide/propylene oxide block alkyl polyamine ethoxylates such as PLURONIC® from BASF; **C14-C22** mid-chain branched alcohols as discussed in US 6,150,322; C_{14}-C22 mid-chain branched alkyl alkoxylates, BAE_x, wherein x is from 1-30, as discussed in US 6,153,577, US 6,020,303 and US 6,093,856

Specific nonionic detergent compounds are C_6 to C22 alkyl phenol-ethylene oxide condensates, generally 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per

molecule, and the condensation products of aliphatic C_8 to C_{18} primary or secondary linear or branched alcohols with ethylene oxide, generally 5 to 40 EO.

Suitable anionic detergent compounds which may be used are usually water-soluble alkali metal salts of organic carboxylates, sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Nonlimiting examples of anionic surfactants useful herein include: C_{9}-C_{18} alkyl benzene sulphonates (LAS); C_{10}-C_{20} primary, branched-chain and random alkyl sulphates (AS); C_{10}-C_{18} secondary (2,3) alkyl sulphates; C_{10}-C_{18} alkyl alkoxy sulphates (AE_XS) wherein preferably x is from 1-30; C_{10}-C_{18} alkyl alkoxy carboxylates preferably comprising 1-5 ethoxy units; mid-chain branched alkyl sulpates as discussed in US 6,020,303 and US 6,060,443; mid-chain branched alkyl alkoxy sulphates as discussed in US 6,008,181 and US 6,020,303; modified alkylbenzene sulphonate (MLAS) as discussed in WO 99/05243, WO 99/05242, and WO 99/05244; methyl ester sulphonate (MES); and alpha-olefin sulfonate (AOS).

The preferred anionic detergent compounds are sodium C_{11} to C_{15} alkyl benzene sulphonates and sodium C_{12} to C_{18} alkyl sulphates. Also applicable are surfactants such as those described in EP-A-328 177 (Unilever), which show resistance to salting-out, the alkyl polyglycoside surfactants described in EP-A-070 074, and alkyl monoglycosides.

Preferred surfactant systems are mixtures of anionic with nonionic detergent active materials, in particular the groups and examples of anionic and nonionic surfactants pointed out in EP-A-346 995 (Unilever). Especially preferred is surfactant system that is a mixture of an alkali metal salt of a C_{16} to C_{18} primary alcohol sulphate together with a C_{12} to C_{15} primary alcohol 3 to 7 EO ethoxylate.

The nonionic detergent is preferably present in amounts greater than 10%, e.g. 25 to 90 wt.% of the surfactant system. Anionic surfactants can be present for example in amounts in the range from about 5 wt.% to about 40 wt.% of the surfactant system.

Methyl ether based surfactants can be used instead of the simple alkyl based surfactants described above.

CATIONIC FABRIC SOFTENING COMPOUND

The cationic surfactant may be a cationic fabric softening compound. Preferred cationic fabric softening compounds are water insoluble quaternary ammonium material which comprises a compound having two C_{12-18} alkyl or alkenyl groups connected to the nitrogen head group via at least one ester link. It is more preferred if the quaternary ammonium material has two or more ester links.

BUILDERS OR COMPLEXING AGENTS

The composition may optionally comprise from 0 to 50 wt.% of a detergency builder. Preferably builder is present at a level of from 1 to 40 wt.%.

Builder materials may be selected from 1) calcium sequestrant materials, 2) precipitating materials, 3) calcium ion-exchange materials and 4) mixtures thereof.

It is preferred that when an insoluble inorganic builder, e.g., zeolite is used, the size is in the range 0.1 to 10 microns (as measured by The Mastersizer 2000 particle size analyzer using laser diffraction ex Malvern™).

Examples of calcium sequestrant builder materials include alkali metal polyphosphates, such as sodium tripolyphosphate and organic sequestrants, such as ethylene diamine tetra-acetic acid.

Examples of precipitating builder materials include sodium orthophosphate and sodium carbonate.

Examples of calcium ion-exchange builder materials include the various types of water-insoluble crystalline or amorphous aluminosilicates, of which zeolites are the best known representatives, e.g. zeolite A, zeolite B (also known as zeolite P), zeolite C, zeolite X, zeolite Y and also the zeolite P-type as described in EP-A-0,384,070.

The composition may also contain 0-50 wt.% of a builder or complexing agent such as ethylenediaminetetraacetic acid, diethylenetriamine-pentaacetic acid, alkyl- or alkenylsuccinic acid, nitrilotriacetic acid or the other builders mentioned below. Many builders are also bleach-stabilising agents by virtue of their ability to complex metal ions.

Zeolite and carbonate (carbonate (including bicarbonate and sesquicarbonate) are preferred builders.

The composition may contain as builder a crystalline aluminosilicate, preferably an alkali metal aluminosilicate, more preferably a sodium aluminosilicate. This is typically present at a level of less than 15 wt.%. Aluminosilicates are materials having the general formula:

$$0.8\text{-}1.5\ M_2O\ .\ Al_2O_3.\ 0.8\text{-}6\ SiO_2$$

where M is a monovalent cation, preferably sodium. These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiO_2 units in the formula above. They can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature. The ratio of surfactants to aluminosilicate (where present) is preferably greater than 5:2, more preferably greater than 3:1 .

Alternatively, or additionally to the aluminosilicate builders, phosphate builders may be used. In this art the term 'phosphate' embraces diphosphate, triphosphate, and phosphonate species. Other forms of builder include silicates, such as soluble silicates, metasilicates, layered silicates (e.g. SKS-6 from Hoechst).

Preferably the laundry detergent formulation is a non-phosphate built or largely un-built laundry detergent formulation, i.e., contains less than 1 wt.% of phosphate.

SHADING AGENT

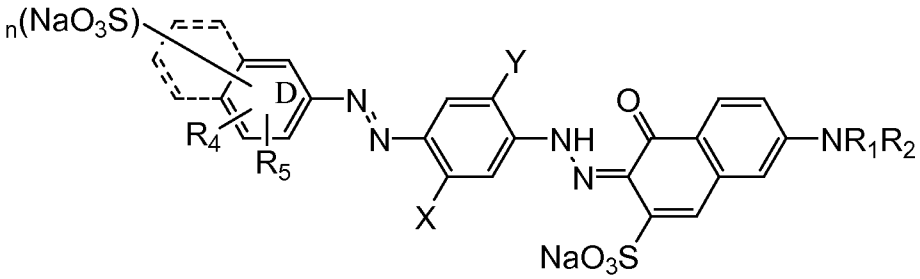
The composition preferably comprises a blue or violet shading agent in the range from 0.0001 to 0.01 wt.%. The shading agents reduce the perception of damage to many coloured garments and increase the perception of whiteness of white garments.

The shading agents are preferably selected from blue and violet dyes of the solvent disperse basic, direct and acid type listed in the colour index (Society of Dyers and Colourists and American Association of Textile Chemists and Colorists 2002).

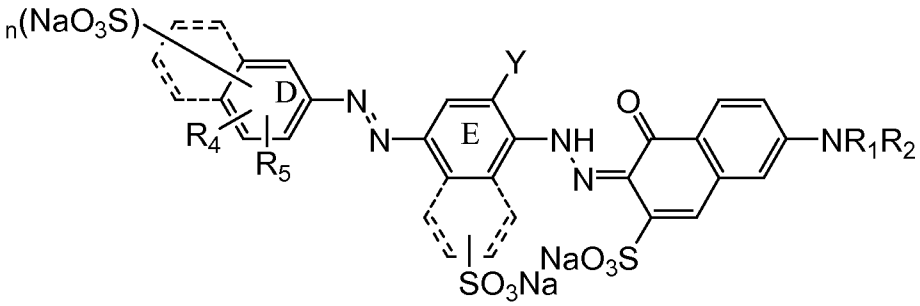
Preferably a direct violet or direct blue dyes is present. Preferably the dyes are *bis-azo, tris-azo* dyes or triphendioxazine dye. The carcinogenic benzidene based dyes are not preferred.

Bis-azo copper containing dyes such as direct violet 66 may be used.

The most preferred bis-azo dyes have the following structure:



or



wherein:

ring D and E may be independently naphthyl or phenyl as shown;

Ri is selected from: hydrogen and C 1-C4-alkyl, preferably hydrogen;

R2 is selected from: hydrogen, C1-C4-alkyl, substituted or unsubstituted phenyl and substituted or unsubstituted naphthyl, preferably phenyl;

R 3 and **R_4** are independently selected from: hydrogen and C1-C4-alkyl, preferably hydrogen or methyl;

X and Y are independently selected from: hydrogen, C1-C4-alkyl and C1-C4-alkoxy; preferably the dye has X= methyl; and, Y = methoxy and n is 0, 1 or 2, preferably 1 or 2.

Preferred bis-azo dyes are direct violet 7, direct violet 9, direct violet 11, direct violet 26, direct violet 31, direct violet 35, direct violet 40, direct violet 41, direct violet 51, and direct violet 99.

Preferred solvent and disperse dyes, are selected from, mono-azo or anthraquinone dyes, most preferably, solvent violet 13, disperse violet 27 disperse violet 26, disperse violet 28, disperse violet 63 and disperse violet 77.

A preferred pigment is pigment violet 23.

Examples of suitable organic dyes are disclosed in WO2008/017570. Preferred dyes also include Acid Violet 50 and Acid Blue 98. Polymeric shading/hueing agents are also preferred.

The shading/hueing agent may be present in the cues and/or in the bulk of the formulation.

ENZYMES

The composition preferably comprises one or more enzymes which provide cleaning performance and/or fabric care benefits. Examples of suitable enzymes include, but are not limited to, hemicellulases, peroxidases, proteases, cellulases, xylanases, lipases, phospholipases, esterases, cutinases, pectinases, mannanases, pectate lyases, keratinases, reductases, oxidases, phenoloxidases, lipoxygenases, ligninases, pullulanases, tannases, pentosanases, malanases, arabinosidases, hyaluronidase, chondroitinase, laccase, and amylases, or

mixtures thereof. A typical combination is an enzyme cocktail that may comprise, for example, a protease and lipase in conjunction with amylase and/or cellulase. When present in a cleaning composition, the aforementioned enzymes may be present at levels from about 0.00001 wt.% to about 2 wt.%, from about 0.0001 wt.% to about 1 wt.% or even from about 0.001 wt.% to about 0.5 wt.% enzyme protein by weight of the composition.

Preferred enzymes are cellulase, lipase, protease and mixtures including one or more of these. The enzymes may be present in the cues, and/or in the bulk of the formulation.

FLUORESCENT AGENT

The composition preferably comprises a fluorescent agent (optical brightener). Fluorescent agents are well known and many such fluorescent agents are available commercially. Usually, these fluorescent agents are supplied and used in the form of their alkali metal salts, for example, the sodium salts. The total amount of the fluorescent agent or agents used in the composition is generally from 0.005 to 2 wt.%, more preferably 0.01 to 0.1 wt.%. Preferred classes of fluorescer are: Di-styryl biphenyl compounds, e.g. Tinopal (Trade Mark) CBS-X, Di-amine stilbene di-sulphonic acid compounds, e.g. Tinopal DMS pure Xtra and Blankophor (Trade Mark) HRH, and Pyrazoline compounds, e.g. Blankophor SN. Preferred fluorescers are: sodium 2-(4-styryl-3-sulfophenyl)-2H-napthol[1 ,2-d]trazole, disodium 4,4'-bis{[(4-anilino-6-(N methyl-N-2 hydroxyethyl) amino 1,3,5-triazin-2-yl)]amino}stilbene-2-2' disulfonate, disodium 4,4'-bis{[(4-anilino-6-morpholino-1 ,3,5-triazin-2-yl)]amino} stilbene-2-2' disulfonate, and disodium 4,4'-bis(2-sulfoslyryl)biphenyl.

The fluorescer may be present in the cues, and/or in the bulk of the formulation.

PERFUME

Preferably the composition comprises a perfume. The perfume is preferably in the range from 0.001 to 3 wt.%, most preferably 0.1 to 1 wt.%. Many suitable examples of perfumes are provided in the CTFA (Cosmetic, Toiletry and Fragrance Association) 1992 International Buyers Guide, published by CFTA Publications and OPD 1993 Chemicals Buyers Directory 80th Annual Edition, published by Schnell Publishing Co.

It is commonplace for a plurality of perfume components to be present in a formulation. In the compositions of the present invention it is envisaged that there will be four or more, preferably five or more, more preferably six or more or even seven or more different perfume components.

In perfume mixtures preferably 15 to 25 wt.% are top notes. Top notes are defined by Poucher (Journal of the Society of Cosmetic Chemists 6(2):80 [1955]). Preferred top-notes are selected from citrus oils, linalool, linalyl acetate, lavender, dihydromyrcenol, rose oxide and cis-3-hexanol.

Perfume and particularly top notes may be used to cue fabric care benefits.

If the laundry treatment composition takes a liquid form, then it is preferred that the composition does not contain a peroxygen bleach, e.g., sodium percarbonate, sodium perborate, and peracid.

POLYMERS

The composition may comprise one or more polymers in addition to any polymer present as the matrix of the elements. Examples are carboxymethylcellulose,

poly(ethylene glycol), polyvinyl alcohol), polycarboxylates such as polyacrylates, maleic/acrylic acid copolymers and lauryl methacrylate/ acrylic acid copolymers.

Polymers present to prevent dye deposition, for example poly(vinylpyrrolidone), poly(vinylpyridine-N-oxide), and poly(vinylimidazole), are preferably absent from the formulation when a shading dye is present.

HYDROTROPE

For compositions in the form of a liquid, it is useful to include a hydrotrope, which prevents liquid crystal formation. The addition of the hydrotrope thus aids the clarity/transparency of the composition and propmotes the visibility of the cues. Suitable hydrotropes include but are not limited to propylene glycol, ethanol, urea, salts of benzene sulphonate, toluene sulphonate, xylene sulphonate or cumene sulphonate. Suitable salts include but are not limited to sodium, potassium, ammonium, monoethanolamine, triethanolamine. Preferably, the hydrotrope is selected from the group consisting of propylene glycol, xylene sulfonate, ethanol, and urea to provide optimum performance. The amount of the hydrotrope is generally in the range of from 0 to 30%, preferably from 0.5 to 30%, more preferably from 0.5 to 30%, most preferably from 1 to 15%.

BLEACH PRECURSORS

Bleach precursors (e.g. tetra-acetylene diamine and sodium percarbonate) cannot be added directly to a liquid formulation as the resulting peracid species would not be stable. By encapsulating the two precursors within the elements forming the cue the generation of peracid can be prevented until the composition is introduced into the main wash.

A particularly preferred embodiment of the invention comprises a packaged surfactant-containing formulation comprising a visual cue, wherein:

a) the surfactant-containing composition is a pourable, liquid or gel, laundry detergent composition; comprising:

i) from 0.05 to 10 wt.% of elements forming a visual cue, and,

ii) from 2 to 70 wt.%, preferably 10 to 30 wt.% of a surfactant selected from an anionic surfactant, a nonionic surfactant, a cationic surfactant, or a mixture thereof.

b) the package is at least in part transparent,

c) the visual cue comprises a plurality of sheet-like elements of dispersed material, having opposed surfaces wherein:

i) the surfaces of the elements are configured such that when an element is located adjacent a flat inner surface of the package, the elements can only contact the inner surface of the packaging over less than 50% of a surface of the element;

ii) the ratio of the average diameter of the elements, measured flat, to the thickness of the elements is greater than 5:1 , preferably greater than 8:1 ;

iii) the average diameter of the elements is in the range 1-10mm, preferably 3-6mm; and;

iv) the average thickness of the elements is in the range 100-2000 microns, preferably 200-500 microns.

In order that the invention may be further and better understood it will be described below with reference to examples.

EXAMPLES:

Example 1a: Preparation of the Hydrophobically Modified Polymeric Material:

A 10wt% solution of PVOH in water was prepared by placing 100g PVOH (Mowiol 20-98 (trade name), ex Kuraray Specialities) and 900g demineralised water into a flask and heating to 70°C. To this, 10ml of hydrochloric acid (36% aqueous solution) was added to catalyse the reaction and then butyraldehyde was added. The mixture was then stirred at 70°C for 5 hours under an inert atmosphere, after which time the heating was stopped and agitation continued for a further 20 hours at room temperature. The reaction mixture was then brought to a pH of 7 using a sodium hydroxide solution.

The resulting solution was precipitated into acetone to yield the acetalised PVOH polymer and washed repeatedly with acetone (500ml) and then water (50ml). It was then dried under vacuum at 70°C overnight to yield a white polymer.

Example 2: Preparation of cue elements:

A solution comprising 3 parts of the hydrophobically modified PVOH prepared according to Example 1, in solution (15% active) was mixed with 7 parts of slurry containing an encapsulated perfume (31 % perfume loading). The solution was used to form a film by pouring it into a Teflon™-film lined polystyrene bioassay tray. One set of samples was dried such that elements suitable for use in cues cut from the film remains flat when placed in surfactant, the other dried such that

the elements curl in surfactant (radius of curvature ~ 8mm). Star-shaped elements were cut from the film using a hand punch.

The difference in drying conditions were, that for the elements which would curve, heat was only applied to one side of the film, whereas those that remain flat were dried by uniform application of heat to the film (hanging in an oven at 75 Celsius for 20 minutes). Uneven heating was accomplished by drying the film on a glass plate heated from underneath (at 75 Celsius for 20 minutes).

Example 3: Evaluation of adhesion:

A glass slide was taken and a small quantity of surfactant-containing product (Persil Small & Mighty) was placed 50mm from one end. A single element of which a plurality would form the visual cue was then placed in the surfactant and left for 10 minutes. The glass slide was then supported at a 40° angle to the horizontal and water at room temperature dripped onto the slide from a 3mm diameter nozzle so that the water ran down over the cue at a rate of 7ml/min using a peristaltic pump. The time taken for the cue element to detach from the slide and fall to the bottom edge was measured. In repeated experiments, the average time for a flat cue element was 902.25 seconds, whereas the average time for a curved cue element was 64.5s.

Example	Release time (comparative) (flat cue element) - sec.	Release time (embodiment) (curved cue element) - sec.
3a	874	75
3b	1072	67
3c	702	60
3d	901	56

These results clearly show that the curved elements are far less liable to adhere to surfaces than flat elements.

CLAIMS

1. A packaged pourable liquid or a gel surfactant-containing formulation comprising a visual cue, wherein the visual cue comprises a plurality of sheet-like elements of dispersed material, having opposed surfaces wherein the surfaces of the elements are configured such that when an element is located adjacent a flat inner surface of the package, the elements can only contact the inner surface of the packaging over less than 50% of a surface of the element.

2. A packaged surfactant-containing formulation according to claim two which has a viscosity at 20 sec^{-1} of from 100 mPa.s to 2000 mPa.s.

3. A packaged surfactant-containing formulation according to any preceding claim wherein the packaging is at least in part transparent.

4. A packaged surfactant-containing formulation according to any preceding claim wherein the ratio of the average diameter of the elements, measured flat, to the thickness of the elements is greater than 5:1 , preferably greater than 8:1 .

5. A packaged surfactant-containing formulation according to any preceding claim wherein the average diameter of the elements is in the range 1-10mm, preferably 3-6mm.

6. A packaged surfactant-containing formulation according to any preceding claim wherein the average thickness of the elements is in the range 100-2000 microns, preferably 200-500 microns.

7. A packaged surfactant-containing fornnulation according to any preceding claim wherein the elements are "star" or "flower"-shaped.

8. A packaged surfactant-containing formulation according to any preceding claim wherein the elements comprise a hydrophobically modified polymer.

9. A packaged surfactant-containing formulation according to claim 8 wherein the elements comprise the hydrophobically modified polymer (i), and a fabric benefit agent (ii), in a ratio of from 1:50 to 99:1 parts by weight, preferably from 1:40 to 95:1 parts by weight.

10. A packaged surfactant-containing formulation according to any of claims 8-9, wherein the elements comprise a hydrophobically modified polymer having a backbone selected from the group consisting of polyvinyl alcohol, polyvinyl acetate, cellulose ethers, polyethylene oxide, starch, polyvinylpyrrolidone, polyacrylamide, polyvinyl methyl ether-maleic anhydride, polymaleic anhydride, styrene maleic anhydride, hydroxyethylcellulose, methylcellulose, polyethylene glycols, carboxymethylcellulose, polyacrylic acid salts, alginates, acrylamide copolymers, guar gum, casein, ethylene-maleic anhydride resin series, polyethyleneimine, ethyl hydroxyethylcellulose, ethyl methylcellulose, hydroxyethyl methylcellulose and copolymers of the monomers thereof.

11. A packaged surfactant-containing formulation according to any preceding claim wherein the elements comprise a matrix material (i), and a fabric benefit agent (ii), in a ratio of from 1:50 to 99:1 parts by weight, preferably from 1:40 to 95:1 parts by weight.

12. A packaged surfactant-containing formulation according to any preceding claim which comprises from 0.05 to 10 wt.% of the elements and from 2 to

70 wt.%, preferably 10 to 30 wt.% of a surfactant selected from an anionic surfactant, a nonionic surfactant, a cationic surfactant, or a mixture thereof.

13. A process for making elements for inclusion in a packaged pourable liquid or gel surfactant-containing formulation as a visual cue comprising the steps of:

(a) providing an aqueous mixture of a hydrophobically modified polymer as defined herein, and a fabric benefit agent;

(b) drying the aqueous mixture to remove whole or part of the water from the aqueous mixture to form a sheet;

(c) forming elements from the sheet in the desired shape and/or size;

and,

(d) optionally, further drying the elements;

wherein, in at least one of the drying steps (b) and (d) one face of the elements is, at least in part, dried more quickly than the other.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/060521

A. CLASSIFICATION OF SUBJECT MATTER
INV. C11D17/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2009/047124 A1 (UNILEVER PLC [GB]; UNILEVER NV [NL]; UNILEVER HINDUSTAN [IN]; CUTRONA) 16 April 2009 (2009-04-16)	13
A	the example; page 26	1-12

X	US 7 375 070 B2 (PEGELOW ULRICH [DE] ET AL) 20 May 2008 (2008-05-20) claims column 2, line 66 - column 3, line 38	1-12

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" documentwhich may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
14 October 2011	25/10/2011

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2011/060521

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