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(54) **Water soluble package and liquid contents thereof**

Wasserlösliche Verpackung und zugehörige Flüssigkeitsinhalte

Emballage hydrosoluble et contenu liquide de celui-ci

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**US-A1- 2002 128 170 US-A1- 2002 187 909**  
**US-B1- 6 335 315**

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**EP 1 400 460 B1**

**Description****Field of the invention**

5 **[0001]** The present invention relates to a water soluble package for containing a liquid cleaning composition, especially a laundry treatment agent. In particular, the invention relates to a water soluble package formed from a water soluble film containing a substantially non-aqueous liquid composition.

**Background of the invention**

10 **[0002]** Water soluble packages are known in the detergent art and generally comprise either vertical form-fill-seal (VFFS) envelopes or thermoformed envelopes. In one of the VFFS processes, a roll of water soluble film is sealed along its edges to form a tube, which tube is heat sealed intermittently along its length to form individual envelopes which are filled with product and heat sealed.

15 The thermoforming process generally involves moulding a first sheet of water soluble film to form one or more recesses adapted to retain a composition, such as for example a solid detergent composition, placing the composition in the at least one recess, placing a second sheet of water soluble material over the first so as to cover the or each recess, and heat sealing the first and second sheets together at least around the recesses so as to form one or more water soluble packages. Solvent-sealed water soluble packages are also known in the art.

20 **[0003]** Cleaning products are traditionally often liquids, viscous or thin, such as known for personal cleaning (bath and shower liquids and shampoos) or for domestic cleaning (hand dish wash and other hard surface cleaning, laundry-cleaning etc.). Other products are solids, such as powders, granules, small capsules (up to 2mm diameter) or more recently tablets, for laundry and machine dish wash, and soap bars for skin cleaning. Recently, so called unit dose products are experiencing an increasing success with consumers, because they eliminate the need for manipulating, and possibly spilling, liquids or powders and simplify the use of a correct dose of the cleaning product for the required purpose. Examples thereof are the laundry and machine dish wash tablets mentioned above.

**Prior art**

30 **[0004]** Many types of water soluble packages are known, including packages made from polyvinyl alcohol (PVOH) film. A wide variety of different materials can be packaged in such films, including liquid materials. EP-A-518689 discloses a containerisation system for hazardous materials (for example pesticides) comprising a PVOH film enclosing a composition comprising the hazardous material, water, an electrolyte and optional other materials. The electrolyte is added to reduce the solubility of the film to prevent its dissolution by the packaged composition.

35 **[0005]** EP-A-700989 discloses a unit packaged detergent for dish washing, the package comprising a detergent composition wrapped in PVOH film, wherein the film protects the detergent from dissolution until the main wash cycle of the dish washing machine.

**[0006]** EP-A-593952 discloses a water soluble sachet of PVOH with two chambers and a treatment agent for washing inside each chamber.

40 **[0007]** EP-A-941939 relates to a water soluble package, which can be PVOH, containing a composition which, when dissolved, produces a solution of known composition.

**[0008]** GB-A-2305931 discloses a dissolvable laundry sachet and BE-9700361 relates to a water soluble unit-dosed cleaning agent, especially for cleaning hands.

**[0009]** A variety of water soluble PVOH films are also known. For example, EP-B-157162 relates to a self-supporting film comprising a PVOH matrix having rubbery microdomains dispersed therein.

45 **[0010]** The specifications of International Patent Applications WO-A-00/55044, WO-A-00/55045, WO-A-00/55046, WO-A-00/55068, WO-A-00/55069 and WO-A-00/55415 disclose water soluble packages containing a fluid substance (defined as a liquid, gel or paste) which is a horizontal form-fill-seal (HFFS) envelope. These packages comprise a body wall portion having internal volume and which is preferably dome-shaped, formed from a first sheet, and a superposed base wall portion, formed from a second sheet, sealed to the body wall portion.

50 **[0011]** When formulating a liquid unit dose product of the kind wherein a substantially non-aqueous formulation is encapsulated in a water soluble film, probably the most difficult challenge is to preserve the physical integrity and stability of the film.

55 One approach to address this problem is disclosed in US-A-4,743,394 and US-A-5,362,413. In these documents, detergent compositions packaged in water soluble films are described. It is further mentioned that plasticizers such as trimethylolpropane, glycerol, polyethylene glycol and others known to those skilled in the art can be included in the film so as to provide the film strength and flexibility required for producing, filling, shipping and storing the pouches prepared from these films. It is also disclosed in these documents that when pouches produced from these plasticizer-

containing films are stored in contact with a detergent composition, significant loss of impact strength can occur, and that such loss of strength can be minimized by incorporation of known plasticizers into the detergent composition itself. Another approach is disclosed in WO-02/060980 referring to rapidly dissolvable polymer films and articles made therefrom, and generally describing that the solvent system used in the film forming composition will preferably have at least one common solvent material as the solvent system used in the encapsulated composition, so as to prevent problems with solvent migration.

**[0012]** However, there still remains a need for further improving the compression strength and integrity of the package formed from the water soluble film and maintaining said strength on storage at all times, particularly when the package is applied for containing substantially non-aqueous liquid detergent.

**[0013]** Therefore, an object of the invention is to find a package formed from a water soluble film and containing a substantially non-aqueous liquid composition, wherein the package has considerably improved compression strength as compared to known packages of the prior art, particularly during prolonged storage periods.

We have now surprisingly found that this and other objects have been achieved by the water soluble package of the present invention.

**[0014]** In particular, it has been unexpectedly found that the compression strength of the water soluble package can be significantly increased, when the encapsulated non-aqueous liquid composition contains at least two different solvents, being a primary and a secondary solvent, whereby the concentration of the secondary solvent is at least 5% by weight of the primary solvent, and whereby the film contains a plasticizer which is the same compound as the secondary solvent.

In the context of the present invention, the compression strength is defined as the strength measured when a package formed from a film and containing a liquid composition is compressed at a uniform rate (e.g. 50 mm/minute) between two metal plates, one of which being connected to a load cell. As the distance between these plates is progressively reduced, the load increases and, ultimately, the package bursts: the load measured (in Newtons) at the moment of bursting is the maximum compression strength of the package. The compression strength may be measured in this way using a Universal Electromechanical Tester, such as Instron ®.

#### **Definition of the invention**

**[0015]** Accordingly, the present invention provides a water soluble package formed from a water soluble film containing a substantially non-aqueous liquid composition, said film comprising a plasticizer system and said liquid composition comprising a surfactant, and at least two different solvents, being a primary and a secondary solvent, wherein the liquid composition contains the secondary solvent in an amount of at least 5% by weight of the primary solvent present therein and wherein the main plasticizer in the film is the same chemical as the secondary solvent.

#### **Detailed description of the invention**

##### *The non-aqueous liquid*

**[0016]** The substantially non-aqueous liquid composition effectively provides a cleaning function when released into the wash liquor. Preferably, it is a laundry treatment agent. The amount of this liquid composition in the package, i.e. the unit dose volume may for example be from 10 ml to 100 ml, e.g. from 12.5 ml to 75 ml, preferably from 15 ml to 60 ml, more preferably from 20 ml to 55 ml.

By "substantially non-aqueous" it is meant that the amount of water in the liquid composition is below the level at which the package would dissolve through contact with its contents. Preferably, the liquid composition comprises no more than 20%, more preferably no more than 15%, still more preferably no more than 10%, by weight of water. The viscosity of the liquid composition is suitably at least 25 mPaS but no more than 10,000 mPaS.

##### *The primary solvent*

**[0017]** The liquid composition of the invention contains at least two different solvents, being a primary and a secondary solvent. Suitable primary solvents include water, alcohols, ethers, polyethers, polyols, alkylamines, alkanol amines and fatty amines, alkyl (or fatty) amides and mono-and di- N-alkyl substituted derivatives thereof, alkyl (or fatty) carboxylic acid lower alkyl esters, ketones, aldehydes, glycerides, and nonionic surfactants such as alkoxylated alcohols. Preferred primary solvents are selected from the group consisting of pentanediols, butanediols, propanediols, such as 1,3-propane diol, alkanol amines, di-alkyl ethers, polyethylene glycols, alkyl ketones (such as acetone) and glyceryl trialkylcarboxylates (such as glyceryl tri-acetate), glycerol, and sorbitol. Even more preferred primary solvents are butanediols, and propanediols. Most preferred primary solvent for use in the composition of the present invention is monopropylene glycol.

**[0018]** Preferably, the primary solvent is present in the liquid composition in an amount of at least 10% by weight, more preferably from 15% to 50% by weight.

*Plasticizer/secondary solvent*

**[0019]** The water soluble film of the invention incorporates at least one plasticizer which is the same compound as the secondary solvent in the liquid composition. This plasticizer needs to be the main plasticizer in the film: it is desirably present in the film at a concentration of at least 10% by weight. Furthermore, it is essential that the secondary solvent is present in the liquid composition in an amount of at least 5%, preferably at least 10%, by weight of the primary solvent present therein.

As will be elucidated in more detail hereinbelow, the water soluble film may be formed from a variety of different materials. The preferred type of plasticizer will depend on the nature of the film in question. Preferred plasticizers are recited in more detail below.

**[0020]** The plasticizer system in the film influences the way the polymer chains usually present in the film 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 consequences of these intrusions and their propensity to revert or recover to their former state. The key feature of preferred plasticisers is that they are highly compatible with the film, and are normally hydrophilic in nature.

The envelope forming the package is preferably formed by horizontal, vertical or rotary form-film-seal techniques.

*The water soluble film*

**[0021]** The film effectively comprises a water soluble polymer.

As used herein, the term "water soluble polymer" refers to a polymer which dissolves and/or dispenses completely in water within 30 minutes with agitation, e.g. by means of hand, stick or other stirrer or under the action of a mechanical washing machine and at a relevant temperature. A "relevant temperature" is one at which the consumer will need to dissolve or disperse the polymer component at the beginning of, or during a cleaning process. A polymer is to be regarded as dissolving or dispersing at a "relevant temperature" if it does so under the aforementioned conditions at a temperature anywhere in the range of from 20°C to 60°C.

**[0022]** Preferred water soluble polymers are those capable of being cast into a film or solid mass and may for example as described in Davidson and Sittig, Water-Soluble Resins, Van Nostrand Reinhold Company, New York (1968). The water-soluble polymer should have proper characteristics, such as strength and heat-sealability, to permit machine handling during the processes of making the water soluble package. Preferred water-soluble resins include polyvinyl alcohol and copolymers thereof, cellulose ethers, polyethylene oxide, polyvinylpyrrolidone, polymaleic anhydride and copolymers thereof, hydroxyethylcellulose, methylcellulose, acrylamide and copolymers thereof, polyethyleneimine, ethyl hydroxyethylcellulose, ethyl methylcellulose, hydroxyethyl methylcellulose. In this context, it is noted that copolymers can be made of 2 or more types of monomers.

**[0023]** Water-soluble, polyvinyl alcohol film-forming resins are most preferred for use in the package of the present invention.

**[0024]** Polyvinyl alcohols (PVA) preferred for use herein have an average molecular weight anywhere between 1,000 and 100,000, preferably between 5,000 and 250,000, for example between 15,000 and 150,000. Hydrolysis, or alcoholysis, is defined as the percent completion of the reaction where acetate groups on the resin are substituted with hydroxyl, -OH, groups. A hydrolysis range of from 60-99% of polyvinyl alcohol film-forming resin is preferred, while a more preferred range of hydrolysis is from about 70-90% for water-soluble, polyvinyl alcohol film-forming resins. The most preferred range of hydrolysis is 80-89%. As used in this application, the term "polyvinyl alcohol" includes polyvinyl acetate compounds with levels of hydrolysis disclosed herein. The water-soluble resin film should be formulated so as to substantially completely dissolve in 50°C. water with agitation within about thirty minutes, preferably within about 15 minutes in 50°C. water with agitation, and most preferably within about 5 minutes in 50°C. water with agitation.

**[0025]** Suitable PVA films for use in a package according to the invention are commercially available and described, for example, in EP-B- 291,198. PVA films for use in a package according to the invention can be made by the copolymerisation of vinyl acetate and a carboxylate-containing monomer (for example acrylic, maleic or itaconic acid or acid ester), followed by partial (for example up to about 90%) hydrolysis with sodium hydroxide.

**[0026]** Suitable water soluble films can also be made from blends of two or more polymers/copolymers as mentioned above, and having different compositions or molecular weights.

**[0027]** Generally speaking, plasticizers suitable for use with PVA-based films have -OH groups in common with the ~CH<sub>2</sub>-CH(OH)-CH<sub>2</sub>-CH(OH)-polymer chain of the film polymer.

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

**[0028]** Water itself is a suitable plasticizer for any of the films recited herein. Preferred main plasticizers are selected from the group consisting of pentane diols, butane diols, propane diols, glycerol, trimethylolpropane, sorbitol, diethylene glycol, triethylene glycol, and dipropylene glycol. The most preferred plasticizer for use as a main plasticizer in the film of the invention is glycerol.

For the sake of clarity, it is noted that this group of preferred main plasticizer compounds is equally preferred for use as a secondary solvent in the liquid composition, and that glycerol is not only the most preferred main plasticizer but also the most preferred type of secondary solvent. It is also noted that the primary and secondary solvents are different chemicals.

**[0029]** The total amount of plasticizer in the film (i.e. per unit weight of film) may vary considerably according to the film type and plasticizer type. It could for example be in the range of from 0.1% to 50%, e.g. 10% to 45%, such as 15% to 40% by weight. In PVA-based films which are preferably used in the present invention, the plasticizer system is desirably present in a total amount of above 15% by weight.

#### Encapsulation methods

##### *(a) Horizontal form-fill-seal*

**[0030]** Water soluble films based on PVA can be made according to any of the horizontal form-fill-seal methods described in any of WO-A-00/55044, WO-A-00/55045, WO-A-00/55046, WO-A-00/55068, WO-A-00/55069 and WO-A-00/55415.

**[0031]** By way of example, a thermoforming process is now described where a number of packages according to the invention are produced from two sheets of water soluble material. Recesses are formed in one of the film sheets using a forming die having a plurality of cavities with dimensions corresponding generally to the dimensions of the packages to be produced. Further, a single heating plate is used for thermoforming the film for all the cavities, and in the same way a single sealing plate is applied.

**[0032]** A first sheet of polyvinyl alcohol film is drawn over a forming die so that the film is placed over the plurality of forming cavities in the die. In this example each cavity is generally dome shape having a round edge, the edges of the cavities further being rounded to remove any sharp edges which might damage the film during the forming or sealing steps of the process. Each cavity further includes a raised surrounding flange. In order to maximise package strength, the film is delivered to the forming die in a crease free form and with minimum tension. A heating plate is used to heat the film, which plate is positioned to superpose the forming die. During this preheating step, a vacuum of 0.5 bar is pulled through the preheating plate to ensure intimate contact between the film and the pre-heating plate, this intimate contact ensuring that the film is heated evenly and uniformly (the extent of the vacuum is dependant of the thermoforming conditions and the type of film used, however in the present context a vacuum of less than 0.6 bar was found suitable). Non-uniform heating results in a formed package having weak spots. In addition to the vacuum, it is possible to blow air against the film to force it into intimate contact with the preheating plate.

**[0033]** The thermoformed film is moulded into the cavities blowing the film off the heating plate and/or by sucking the film into the cavities thus forming a plurality of recesses in the film which, once formed, are retained in their thermoformed orientation by the application of a vacuum through the walls of the cavities. This vacuum is maintained at least until the packages are sealed. Once the recesses are formed and held in position by the vacuum, a liquid composition according to the invention is added to each of the recesses. A second sheet of polyvinyl alcohol film is then superposed on the first sheet across the filled recesses and heat-sealed thereto using a sealing plate. The raised flanges surrounding each cavity ensure that the films are sealed together along the flange to form a continuous seal. The rounded edge of each cavity is at least partly formed by a resiliently deformable material, such as for example silicone rubber. This results in reduced force being applied at the inner edge of the sealing flange to avoid heat/pressure damage to the film.

**[0034]** Once sealed, the packages formed are separated from the web of sheet film using cutting means. At this stage it is possible to release the vacuum on the die, and eject the formed packages from the forming die. In this way the packages are formed, filled and sealed while nesting in the forming die. In addition they may be cut while in the forming die as well.

**[0035]** During the forming, filling and sealing steps of the process, it may be desirable to maintain the relative humidity at a reasonable level. This is done to maintain the heat sealing characteristics of the film. When handling thinner films, it may be necessary to reduce the relative humidity to ensure that the films have a relatively low degree of plasticisation and are therefore stiffer and easier to handle.

##### *(b) Vertical Form-Fill-Seal*

**[0036]** In the vertical form-fill-seal (VFFS) technique, a continuous tube of flexible plastics film is extruded. It is sealed,

preferably by heat or ultrasonic sealing, at the bottom, filled with the liquid composition, sealed again above the liquid film and then removed from the continuous tube, e.g. by cutting.

*(c) Rotary Form-Fill-Seal*

**[0037]** (d) Alternatively, a rotary form-fill-seal technique may be used. In this technique, forming, filling and sealing of water soluble packages is carried out using a rotary drum that has forming cavities or recesses on its curved surface.

**[0038]** Instead of heat sealing as described above, solvent sealing, ultrasonic sealing or any other type of sealing known in the art could be applied for producing the package of the present invention. When solvent sealing is used and the film contains PVA, an aqueous solution is preferably used as a solvent.

*Surfactant material*

**[0039]** The surfactant present in the liquid composition may be selected from nonionic, anionic, cationic and amphoteric detergent surfactants. These may be in liquid form or as solid dissolved or dispersed in the substantially non-aqueous liquid composition.

**[0040]** Nonionic detergent surfactants are well-known in the art. They normally consist of a water-solubilizing polyalkoxylene or a mono- or di-alkanolamide group in chemical combination with an organic hydrophobic group derived, for example, from alkylphenols in which the alkyl group contains from about 6 to about 12 carbon atoms, dialkylphenols in which primary, secondary or tertiary aliphatic alcohols (or alkyl-capped derivatives thereof), preferably having from 8 to 20 carbon atoms, monocarboxylic acids having from 10 to about 24 carbon atoms in the alkyl group and polyoxypropylene. Also common are fatty acid mono- and dialkanolamides in which the alkyl group of the fatty acid radical contains from 10 to about 20 carbon atoms and the alkyl group having from 1 to 3 carbon atoms. In any of the mono- and di-alkanolamide derivatives, optionally, there may be a polyoxyalkylene moiety joining the latter groups and the hydrophobic part of the molecule. In all polyalkoxylene containing surfactants, the polyalkoxylene moiety preferably consists of from 2 to 20 groups of ethylene oxide or of ethylene oxide and propylene oxide groups. Amongst the latter class, particularly preferred are those described in European specification EP-A-225,654. Also preferred are those ethoxylated nonionics which are the condensation products of fatty alcohols with from 9 to 15 carbon atoms condensed with from 3 to 11 moles of ethylene oxide. Examples of these are the condensation products of C<sub>11-13</sub> alcohols with (say) 3 or 7 moles of ethylene oxide. These may be used as the sole nonionic surfactants or in combination with those of the described in the last-mentioned European specification. These nonionics may also be suitably used as primary solvent material.

*Anionic surfactants*

**[0041]** In addition, the liquid compositions of the invention may also comprises an anionic surfactant. Preferred anionic surfactants are the linear alkyl benzene sulfonate (LAS) materials. Such surfactants and their preparation are described for example in U.S. Patents 2,220,099 and 2,477,383, incorporated herein by reference. Particularly preferred are the sodium, potassium and mono-, di- or tri-ethanolammonium linear straight chain alkylbenzene sulfonates in which the average number of carbon atoms in the alkyl group is from about 11 to 14.

**[0042]** Monoethanol ammonium salt of C<sub>11</sub>-C<sub>14</sub>, e.g., C<sub>12</sub>, LAS is especially preferred. Preferred anionic surfactants include the alkyl sulfate surfactants hereof being water soluble salts or acids of the formula ROSO<sub>3</sub>M wherein R preferably is a C<sub>10</sub>-C<sub>24</sub> hydrocarbyl, preferably an alkyl or hydroxyalkyl having a C<sub>10</sub>-C<sub>18</sub> alkyl component, more preferably a C<sub>12</sub>-C<sub>15</sub> alkyl or hydroxyalkyl, and M is H or a cation, e.g., an alkali metal cation (e.g. sodium, potassium, lithium), or ammonium or substituted ammonium, especially mono-di-, or tri-ethanolammonium.

**[0043]** Preferred anionic surfactants include alkyl alkoxylated sulfate surfactants hereof being water soluble salts or acids of the formula RO(A)<sub>m</sub>SO<sub>3</sub>M wherein R is an unsubstituted C<sub>10</sub>-C<sub>24</sub> alkyl or hydroxyalkyl group having a C<sub>10</sub>-C<sub>24</sub> alkyl component, preferably a C<sub>12</sub>-C<sub>18</sub> alkyl or hydroxyalkyl, more preferably C<sub>12</sub>-C<sub>15</sub> alkyl or hydroxyalkyl, A is an ethoxy or propoxy unit, m is greater than zero, typically between about 0.5 and about 6, more preferably C<sub>12</sub>-C<sub>15</sub> alkyl or hydroxyalkyl, A is an ethoxy or propoxy unit, m is greater than zero, typically between about 0.5 and about 6, more preferably between about 0.5 and about 3, and M is H or a cation which can be, for example, a metal cation (e.g., sodium, potassium, lithium, calcium, magnesium, etc.), ammonium or substituted-ammonium cation such as mono-, di- or tri-ethanolammonium.

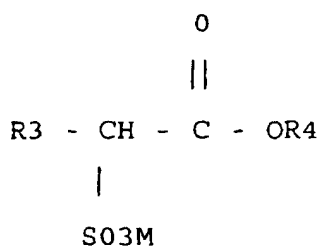
Alkyl ethoxylated sulfates as well as alkyl propoxylated sulfates are contemplated herein. Specific examples of substituted ammonium cations include quaternary ammonium cations such as tetra methyl-ammonium and dimethyl piperdinium cations Exemplary surfactants are C<sub>12</sub>-C<sub>15</sub> alkyl polyethoxylate (1.0) sulfate (C<sub>12</sub>-C<sub>15</sub>E(1.0)M), C<sub>12</sub>-C<sub>15</sub> alkyl polyethoxylate (2.25) sulfate (C<sub>12</sub>-C<sub>15</sub>E(2.25)M), C<sub>12</sub>-C<sub>15</sub> alkyl polyethoxylate (3.0) sulfate (C<sub>12</sub>-C<sub>15</sub>E(3.0)M), and C<sub>12</sub>-C<sub>15</sub> alkyl polyethoxylate (4.0) sulfate (C<sub>12</sub>-C<sub>15</sub>E(4.0)M), wherein M is conveniently selected from sodium,

potassium and mono- di- or tri-ethanolammonium.

One preferred class of anionic surfactants comprises alkylbenzenes sulfonic acids or the alkali salts thereof whereby the alkylbenzenes are alkylated using HF as alkylation catalyst.

**[0044]** Other suitable anionic surfactants to be used are alkyl ester sulfonate surfactants including linear esters of C8-C20 carboxylic acids (i.e., fatty acids) which are sulfonated with gaseous SO<sub>3</sub> according to "The Journal of the American Oil Chemists Society", 52 (1975), pp. 323-329. Suitable starting materials would include natural fatty substances as derived from tallow, palm oil, etc.

**[0045]** The preferred alkyl ester sulfonate surfactant, comprise alkyl ester sulfonate surfactants of the structural formula:



wherein R3 is a C8-C20 hydrocarbonyl, preferably an alkyl, or combination thereof, R4 is a C1-C6 hydrocarbonyl, preferably an alkyl, or combination thereof, and M is a cation which forms a water soluble salt with the alkyl ester sulfonate. Suitable salt-forming cations include metals such as sodium, potassium, and lithium, and substituted or unsubstituted ammonium cations such as mono-, di-, or tri-ethanolammonium.

Preferably, R3 is C10-C16 alkyl, and R4 is methyl, ethyl or isopropyl.

Especially preferred are the methyl ester sulfonates wherein R3 is C10-C16 alkyl.

**[0046]** Other anionic surfactants useful for detergent purposes may also be included in the laundry detergent compositions of the present invention.

**[0047]** These may include salts, for example, sodium, potassium, ammonium, and substituted ammonium salts (such as mono-, di- and triethanolamine salts) of soap, C9-C20 linear alkylbenzenesulfonates, C8-C22 primary or secondary alkanesulfonates, C8-C24 olefinsulfonates, sulfonated polycarboxylic acids prepared by sulfonation of the pyrolyzed product of alkaline earth metal citrates, e.g., as described in British patent specification No. 1,082,179, C8-C24 alkyl-polyglycol ethersulfates (containing up to 10 moles of ethylene oxide); alkyl glycerol sulfonates, fatty acyl glycerol sulfonates, fatty oleyl glycerol sulfates, alkyl phenol ethylene oxide ether sulfates, paraffin sulfonates, alkyl phosphates, isethionates such as the acyl isethionates, N-acyl taurates, alkyl succinamates and sulfosuccinates, monoesters of sulfosuccinates (especially saturated and unsaturated C12-C18 monoesters) and diesters of sulfosuccinates (especially saturated and unsaturated C6-C12 diesters), sulfates of alkylpolysaccharides such as the sulfates of alkylpolyglucoside (the nonionic nonsulfated compounds being described below), and alkyl polyethoxy carboxylates such as those of the formula RO(CH<sub>2</sub>CH<sub>2</sub>O)<sub>k</sub>-CH<sub>2</sub>COO-M<sup>+</sup> wherein R is a C8-C22 alkyl, k is an integer from 1 to 10, and M is a soluble salt-forming cation. Resin acids and hydrogenated resin acids are also suitable, such as rosin, hydrogenated rosin, and resin adds and hydrogenated resin acids present in or derived from tall oil.

**[0048]** Further examples are described in "Surface Active Agents and Detergents" (Vol. I and II by Schwartz, Perry and Berch). A variety of such surfactants are also generally disclosed in U.S. Patent 3,929,678, issued December 30, 1975 to Laughn, et al. at Column 23, line 58 through Column 29, line 23.

**[0049]** When included therein, the liquid compositions of the present invention typically comprise from about 1% to about 40%, preferably from about 10% to about 25% by weight of such anionic surfactants.

When present, the anionic surfactants may be incorporated in free acid and/or neutralised form.

#### *Fatty acids*

**[0050]** The liquid composition of the invention may also comprise fatty acids as anionic surfactant component. Examples of fatty acids suitable for use in the present invention include pure or hardened fatty acids derived from palmoleic, safflower, sunflower, soybean, oleic, linoleic, linolenic, ricinoleic, rapeseed oil or mixtures thereof. Mixtures of saturated and unsaturated fatty acids can also be used herein.

**[0051]** It will be recognised that the fatty acid will be present in the liquid detergent composition primarily in the form of a soap. Suitable cations include, sodium, potassium, ammonium, monoethanol ammonium, diethanol ammonium, triethanol ammonium, tetraalkyl ammonium, e.g., tetra methyl ammonium up to tetradecyl ammonium etc. cations.

**[0052]** The amount of fatty acid will vary depending on the particular characteristics desired in the final liquid composition of the invention.

When present, the level of the fatty acid mixture is suitably from 0.1 % to 30%, preferably from 0.5% to 25%, more preferably from 10-20% by weight of the detergent composition.

#### *Other components*

**[0053]** The substantially non-aqueous liquid cleaning composition may further comprise one or more ingredients selected from builders, polymers, fluorescers, enzymes, silicone foam control agents, perfumes, dyes, bleaches and preservatives.

Some of these materials will be solids which are insoluble in the substantially non-aqueous liquid medium. In that case, they will be dispersed in the substantially non-aqueous liquid medium and may be deflocculated by means of one or more acidic components such as selected from inorganic acids, anionic surfactant acid precursors and Lewis acids, as disclosed in EP-A-266,199.

**[0054]** The water soluble film may further comprise the following minor ingredients: anti-blocking agents, such as silica, fillers (e.g. starch and talc), colourants, release agents and surfactants.

**[0055]** The invention will now be illustrated with reference to the following example, in which parts and percentages are by weight.

#### **EXAMPLES 1 and A**

**[0056]** The following non-aqueous liquid detergent compositions were prepared:

| composition no.                  | A               | 1               |
|----------------------------------|-----------------|-----------------|
| Ingredient:                      |                 |                 |
| nonionic surfactant              | 20%             | 20%             |
| anionic surfactant               | 37%             | 37%             |
| ethanol amine                    | 10%             | 10%             |
| monopropylene glycol             | 23%             | 17%             |
| glycerol                         | -               | 6%              |
| polymers                         | 1%              | 1%              |
| phosphonate                      | 1%              | 1%              |
| enzymes                          | 1%              | 1%              |
| colourants, brighteners, perfume | 1%              | 1%              |
| water                            | balance to 100% | balance to 100% |

**[0057]** Both non-aqueous liquid compositions were encapsulated in a PVA-based water soluble film containing a plasticizer system and including glycerol, as the main plasticizer ingredient, at a level of 15% by weight of the film.

**[0058]** It follows that the thus-obtained capsule containing composition 1 is according to the present invention whereas the other capsule containing composition A is not.

**[0059]** These capsules were stored at 20°C and 65% relative humidity for 4 weeks.

The maximum compression strength of both capsules was measured several times during this storage period, using an Instron® Universal Electrochemical Tester. The following result was obtained: while the initial compression strength of both capsules was the same, the decrease in compression strength of the capsule according to the invention was significantly smaller at any time during the storage period of 4 weeks, than the corresponding decrease of the capsule containing composition A.

These results are shown in Figure 1 wherein the change in maximum compression strength as a function of the storage period is depicted for the above-identified capsules. In this Figure, the curve indicated as "6% glycerol" was obtained for the above capsule according to the invention, whereas the curve indicated as "0% glycerol" is for the above capsule according to the prior art. It can be clearly noticed in this Figure that the strength of the capsule of the invention is consistently higher except during the first couple of days of storage.

#### **Claims**

1. A water soluble package formed from a water soluble film containing a substantially non-aqueous liquid compo-

sition, said film comprising a plasticizer system and said liquid composition comprising a surfactant, and at least two different solvents being a primary and a secondary solvent,

**characterized in that** the liquid composition contains the secondary solvent in an amount of at least 5% by weight of the primary solvent present therein and wherein the main plasticizer in the film is the same chemical as the secondary solvent.

2. A water soluble package according to claim 1, wherein the primary solvent is selected from the group consisting of water, butane diols, pentane diols, propane diols, alkanol amines, di-alkyl ethers, polyethylene glycols, alkyl ketones, glyceryl trialkyl carboxylates, monopropylene glycol, sorbitol, and nonionic surfactants.
3. A water soluble package according to claim 2, wherein the primary solvent is selected from the group consisting of butanediols and propanediols.
4. A water soluble package according to claim 3, wherein the primary solvent is monopropylene glycol.
5. A water soluble package according to any one of claims 1-3, wherein the primary solvent is present in the liquid composition in an amount of at least 10% by weight, preferably from 15 to 50% by weight.
6. A water soluble package according to any one of claims 1-5, wherein the main plasticizer is selected from the group consisting of glycerol, pentanediols, butanediols, propanediols, trimethylolpropane, sorbitol, diethylene glycol, triethylene glycol, and dipropylene glycol.
7. A water-soluble package according to claim 6, wherein the main plasticizer is glycerol.
8. A water soluble package according to any one of claims 1-7, wherein the liquid composition contains the secondary solvent in an amount of at least 10% by weight of the primary solvent present therein.
9. A water soluble package according to any one of claims 1-8, wherein the water soluble film comprises film forming polyvinyl alcohol resins.
10. A water soluble package according to any one of claims 1-9, wherein the package is made by a thermoforming process using heat sealing.
11. A water soluble package according to any one of claims 1-9, wherein the package is made by a thermoforming process using solvent sealing.
12. A water soluble package according to any one of claims 1-11, wherein the liquid composition is a laundry treatment agent.

#### Patentansprüche

1. In Wasser lösliche, aus einer in Wasser löslichen Folie gebildete Verpackung, die eine im Wesentlichen nichtwässrige flüssige Zusammensetzung enthält, wobei die Folie ein Weichmachersystem umfasst und die flüssige Zusammensetzung ein Tensid und mindestens zwei verschiedene Lösungsmittel umfasst, und wobei diese Lösungsmittel ein primäres und ein sekundäres Lösungsmittel darstellen, **dadurch gekennzeichnet, dass** die flüssige Zusammensetzung das sekundäre Lösungsmittel in einer Menge von mindestens 5 Gewichtsprozent des darin vorliegenden primären Lösungsmittels enthält und wobei der Hauptweichmacher in der Folie die gleiche Chemikalie wie das sekundäre Lösungsmittel darstellt.
2. In Wasser lösliche Verpackung nach Anspruch 1, wobei das primäre Lösungsmittel aus der Gruppe, bestehend aus Wasser, Butandiolen, Pentandiolen, Propandiolen, Alkanolaminen, Dialkylethern, Polyethylenglycolen, Alkylketonen, Glyceryltrialkylcarboxylaten, Monopropylenglycol, Sorbit und nichtionischen Tensiden, ausgewählt ist.
3. In Wasser lösliche Verpackung nach Anspruch 2, wobei das primäre Lösungsmittel aus der Gruppe, bestehend aus Butandiolen und Propandiolen, ausgewählt ist.
4. In Wasser lösliche Verpackung nach Anspruch 3, wobei das primäre Lösungsmittel Monopropylenglycol darstellt.

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5. In Wasser lösliche Verpackung nach einem der Ansprüche 1 bis 3, wobei das primäre Lösungsmittel in der flüssigen Zusammensetzung in einer Menge von mindestens 10 Gewichtsprozent, vorzugsweise 15 bis 50 Gewichtsprozent, vorliegt.
- 5 6. In Wasser lösliche Verpackung nach einem der Ansprüche 1 bis 5, wobei der Hauptweichmacher aus der Gruppe, bestehend aus Glycerin, Pentandiolen, Butandiolen, Propandiolen, Trimethylolpropan, Sorbit, Diethylenglycol, Triethylenglycol und Dipropylenglycol, ausgewählt ist.
- 10 7. In Wasser lösliche Verpackung nach Anspruch 6, wobei der Hauptweichmacher Glycerin darstellt.
8. In Wasser lösliche Verpackung nach einem der Ansprüche 1 bis 7, wobei die flüssige Zusammensetzung das sekundäre Lösungsmittel in einer Menge von mindestens 10 Gewichtsprozent des darin vorliegenden primären Lösungsmittels enthält.
- 15 9. In Wasser lösliche Verpackung nach einem der Ansprüche 1 bis 8, wobei die in Wasser lösliche Folie filmbildende Polyvinylalkoholharze umfasst.
10. In Wasser lösliche Verpackung nach einem der Ansprüche 1 bis 9, wobei die Verpackung durch ein Warmformverfahren unter Verwendung von Heißsiegeln hergestellt ist.
- 20 11. In Wasser lösliche Verpackung nach einem der Ansprüche 1 bis 9, wobei die Verpackung durch ein Warmformverfahren unter Verwendung von Lösungsmittelsiegeln hergestellt ist.
- 25 12. In Wasser lösliche Verpackung nach einem der Ansprüche 1 bis 11, wobei die flüssige Zusammensetzung ein Wäschebehandlungsmittel darstellt.

### Revendications

- 30 1. Emballage soluble dans l'eau formé d'un film soluble dans l'eau contenant une composition liquide sensiblement non aqueuse, ledit film comprenant un système de plastifiant et ladite composition liquide comprenant un agent tensioactif, et au moins deux solvants différents qui sont un solvant primaire et un solvant secondaire, **caractérisé en ce que** la composition liquide contient le solvant secondaire en une quantité d'au moins 5 % en poids du solvant primaire qui y est présent et dans lequel le plastifiant principal dans le film est le même produit chimique que le solvant secondaire.
- 35 2. Emballage soluble dans l'eau selon la revendication 1, dans lequel le solvant primaire est choisi dans le groupe consistant en l'eau, les butane diols, les pentane diols, les propane diols, les alcanolamines, les dialkyl éthers, les polyéthylène glycols, les alkyl cétones, les trialkyl carboxylates de glycérile, le monopropylène glycol, le sorbitol et les agents tensioactifs non ioniques.
- 40 3. Emballage soluble dans l'eau selon la revendication 2, dans lequel le solvant primaire est choisi dans le groupe consistant en les butane diols et les propane diols.
- 45 4. Emballage soluble dans l'eau selon la revendication 3, dans lequel le solvant primaire est le monopropylène glycol.
5. Emballage soluble dans l'eau selon l'une quelconque des revendications 1 à 3, dans lequel le solvant primaire est présent dans la composition liquide en une quantité d'au moins 10 % en poids, de préférence de 15 à 50 % en poids.
- 50 6. Emballage soluble dans l'eau selon l'une quelconque des revendications 1 à 5, dans lequel le plastifiant principal est choisi dans le groupe consistant en le glycérol, les pentane diols, les butane diols; les propane diols, le triméthylolpropane, le sorbitol, le diéthylène glycol, le triéthylène glycol et le dipropylène glycol.
- 55 7. Emballage soluble dans l'eau selon la revendication 6, dans lequel le plastifiant principal est le glycérol.
8. Emballage soluble dans l'eau selon l'une quelconque des revendications 1 à 7, dans lequel la composition liquide contient le solvant secondaire en une quantité d'au moins 10 % en poids du solvant primaire qui y est présent.

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9. Emballage soluble dans l'eau selon l'une quelconque des revendications 1 à 8, dans lequel le film soluble dans l'eau comprend les résines de polyalcool vinylique filmogènes.

5 10. Emballage soluble dans l'eau selon l'une quelconque des revendications 1 à 9, dans lequel l'emballage est fabriqué par un procédé de thermoformage utilisant un thermoscellage.

11. Emballage soluble dans l'eau selon l'une quelconque des revendications 1 à 9, dans lequel l'emballage est fabriqué par un procédé de thermoformage utilisant un scellage par solvant.

10 12. Emballage soluble dans l'eau selon l'une quelconque des revendications 1 à 11, dans lequel la composition liquide est un agent de traitement lessiviel.

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Fig.1.

