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(54) EXTERNALLY STRUCTURED AQUEOUS ISOTROPIC LIQUID DETERGENT COMPOSITIONS

EXTERN STRUKTURIERTE, WÄSSRIGE, ISOTROPE FLÜSSIGWASCHMITTELZUSAMMENSETZUNGEN

COMPOSITIONS DÉTERGENTES, LIQUIDES, ISOTROPES, AQUEUSES, STRUCTURÉES DE FAÇON EXTERNE

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Description

Technical Field of the Invention

[0001] The present invention relates to externally structured aqueous isotropic liquid detergent compositions comprising surfactant and an external structurant which consists essentially of citrus fibre.

Background of the Invention

[0002] It is desirable to include particulate materials in liquid detergent compositions; for example encapsulated perfumes or visual cues. In addition, it is desirable for liquid detergent compositions to possess rheological properties that provide a sufficient yield stress (also known as critical stress) to ensure that the particulate materials remain stably suspended and dispersed within the composition.

[0003] Isotropic liquid detergent compositions have no innate ability to suspend solid particles, for example visual cues and encapsulates. Whilst it is possible to obtain a suspending medium by appropriate manipulation of the surfactant and electrolyte levels, this imposes undesirable constraints on the composition. This has lead to the use of so-called external 'structurants', to achieve the required rheology and suspending duty without imposing such constraints on the composition. There are numerous such external structurants taught for use in detergent applications.

[0004] The term 'external' used herein in relation to structurants refer to reagents which may be added to a composition whilst maintaining the desired fluid properties of a detergent liquid. This is in contrast to 'internal' structurants which structure detergent liquids from within using, for example, electrolytes or salts and which lead to the formation of a lamellar phase or "gel" which may suspend solid particles. Whilst the use of internal structurants may be a cost effective way of creating suspending properties for particles within a detergent, such compositions often possess a viscosity which is too high, resulting in the need for additional rheology modifiers. It is therefore often preferred to use reagents which may be added to a composition "externally" whilst maintaining the fluid properties.

[0005] There are many structurants employed in detergent applications. One example of a structuring system for suspending solid particles is a combination of clay and a rheology modifying polymer, as disclosed in EP1402877 (Rohm and Haas) and Research Disclosure, June 2000, No. 434, pages 1032-1033.

[0006] Clay has been used in detergent compositions in liquid form as clay sol. In clay sol the clay is pre-dispersed and usually stabilized with additional ingredients proprietary to suppliers. Clay sol is very expensive, which raises the cost of any detergent composition prepared using it. In addition, if clay sol is incorporated into a detergent composition, a preservative is also required as the clay is in liquid form, leading to further cost.

[0007] A further disadvantage of using clays for external structuring in detergent compositions is that they are prone to interaction with other ingredients, with the result that the viscosity of the composition changes over time and syneresis may occur.

[0008] Another structurant employed in detergent compositions is hydrogenated castor oil (HCO) which may be converted into an external structurant by crystallizing it in the composition or in part of the composition. However, the crystallization process often leads to formulation constraints, especially when using high surfactant levels. In addition, HCO structured liquids are often slightly cloudy, which is undesirable for laundry liquid detergents when visual cues are suspended in the liquid. Furthermore, because HCO structurant is formed by cooling, if the detergent composition is subjected to extreme temperatures, a decrease in performance is sometimes observed. Finally, it has been noted that when encapsulated perfume microcapsules are added to concentrated surfactant solutions comprising HCO, the microcapsules may undergo agglomeration into clumps which remain un-dispersed in the liquid detergent. This increases the visibility of the microcapsules and also leads to uneven dosing of the microcapsule contents per wash as the liquid composition is used by a consumer;

[0009] An alternative structurant employed in detergent compositions is citrus fibre. Compositions comprising citrus fibre and the use of same in foodstuffs and personal care compositions are described in US2004/0086626 and US2009/269376.

[0010] Using citrus fibre as a structurant in structured liquid detergents offers the advantage that the citrus fibre is compatible with cleaning and care enzymes, as described in WO2012/052306. The use of citrus fibre in combination with a cationic deposition polymer (Jaguar quaternised guar gum) for anti-dandruff shampoo is disclosed in WO2012/019934, and US 7981855 discloses personal care liquid surfactant compositions comprising up to 15 wt% surfactant, including at least 1 wt% anionic surfactant, and up to 2 wt% bacterial cellulose (preferably MFC). Citrus fibres are also included from 0.001 to 5 wt% to modify the rheological effects of the bacterial cellulose.

[0011] WO 2013/160024 (Unilever) discloses an externally structured aqueous isotropic detergent liquid comprising: a) at least 10 wt percent water, b) at least 3 wt percent mixed detersive surfactant comprising anionic surfactant, c) at least 0.025 wt percent of activated citrus fibre external structurant, characterised in that the liquid further comprises at least 0.1 wt percent of water swellable polyacrylate thickening polymer and the viscosity of the liquid at 20 s -1 and 25

degrees centigrade is at least 0.3 Pa.s. Whilst citrus fibre is a useful structurant for liquid detergent compositions, it is typically known to use it in combination with other structurants such as clays, or alternatively, to use it at high levels to ensure the required rheological and suspending properties are achieved for liquid detergent compositions. However, as well as the associated cost, formulators are aware of the problems which may sometimes be encountered when using combinations of structurants, for example when using clay and polymer combinations leads to syneresis as detailed above. Combinations of structurants are therefore viewed cautiously and are assessed carefully, due to potential undesirable interactions between structurant and other ingredients of the composition.

[0012] It has not previously been possible to use citrus fibre at low levels as the main or sole structurant in externally structured liquid detergents compositions, whilst still providing the required structurant stability and without over-processing the citrus fibres. The use of citrus fiber at low levels without a second structurant, such as clay, in liquid detergent compositions has typically lead to failure of the external structuring system, evidenced by formation of a multi-layer system within the composition and the appearance of a clear unstructured layer forming at the top of containers containing the detergent liquid.

[0013] In order to reduce the amount of citrus fibre in a liquid detergent composition and thereby reduce the cost of preparing same, as well as overcoming problems associated with current detergent compositions comprising multiple and often costly structurants; which potentially react adversely, the inventors have now devised a novel liquid detergent formation which provides the required properties of an effective structurant system and detergent composition without structurant system breakdown.

[0014] It an object of the present invention to provide a process for preparing an improved aqueous isotropic liquid detergent composition with a low level of citrus fibre as the sole active structurant which overcomes the problems associated with known compositions.

[0015] It is also an object of the present invention to provide an improved externally structured aqueous isotropic liquid detergent composition with citrus fibre as the sole active structurant, the composition being obtainable by the inventive process.

Summary of the Invention

[0016] According to a first aspect of the present invention there is provided a process to manufacture an externally structured aqueous liquid detergent composition having a yield stress in the range of 0.3 Pa to 1.2 Pa, the composition comprising at least 10 wt% water; at least 3 wt% detersive surfactant; at least 0.01 wt% non-clay particles; and consisting essentially of 0.05 to 0.125 wt% citrus fibre as sole external structurant comprising the steps of:

a) subjecting citrus fibre to mechanical processing comprising application of shear in the presence of 20 to 120 times the amount of water based on the citrus fibres, the shear being sufficient to cause structural disruption and hydration of the citrus fibres to form a structuring premix comprising dispersed pulped citrus fibre and having a yield stress in the range of 80 to 250 Pa, preferably 80 to 180 Pa;
b) combining and further dispersing the structuring premix formed in step (a) and having a yield stress in the range of 80 to 250 Pa, preferably 80 to 180 Pa with detergent liquid components in the absence of any other external structurants to form the aqueous externally structured liquid detergent composition with a yield stress in the range of 0.3 to 1.2 Pa when measured at 25 °C.

[0017] The detergent composition prepared in accordance with this process is able to successfully suspend non-clay particles, present in the composition without the loss of stability that would be expected from use of such low levels of citrus fibre as the main external structurant in prior art processes. That is, the obtained detergent composition comprises citrus fibre as the sole external structurant and use of material with the specified yield stress of a 2 wt% mixture with water provides sufficient suspending capabilities to enable the citrus pulp to be used as sole external structurant at these low inclusion levels without loss of stability or suspending properties.

[0018] Preferably the detergent composition formed is a laundry detergent composition.

[0019] The composition may advantageously comprise at least 0.1 wt% non-clay particles, more preferably the composition of the present invention comprises at least 0.2 wt% non-clay particles. These particles are stably suspended in the external structured composition. The non-clay particles may be selected from the group comprising: visual cues, perfume encapsulates, care additives and cleaning ingredients. Most preferably, the non-clay particles are perfume encapsulates.

[0020] According to a second aspect of the invention there is provided an externally structured aqueous isotropic liquid detergent composition having a yield stress in the range of 0.3 Pa to 1.2 Pa at 25°C, comprising:

i) at least 10 wt% water;
ii) at least 3 wt% detersive surfactant;

iii) at least 0.01 wt% non-clay particles; and
iv) an external structurant,

characterised in that the external structurant consists essentially of from 0.05 to 0.125 wt% citrus fibre and the composition is obtainable by the process according to the first aspect.

[0021] The yield stress of the detergent composition obtainable according to the present invention is preferably in the range of from 0.30 Pa to 1.0 Pa when measured at 25 °C.

[0022] More preferably the yield stress of the composition according to the present invention is preferably in the range of from 0.35 Pa to 0.85 Pa when measured at 25 °C.

[0023] The citrus fibre is preferably present in the detergent composition in a range of from 0.06 wt% to 0.10 wt%. More preferably, the citrus fibre is present in the composition in a range of from 0.06 wt% to 0.09 wt%. Even more preferably the citrus fibre is present in the composition in a range of from 0.07 wt% to 0.08 wt%.

[0024] Typically the citrus fibre is made up as a premix of citrus fibres that have been hydrated and then "activated" by passing through a HPH (high pressure homogeniser at 500 BarG or about 50,100,000 Pa. A convenient level of citrus fibre in the premix is 2 wt%. Then the citrus fibre premix comprises a yield stress in the range 80 Pa to 160 Pa when measured at 25 °C, More preferably the citrus fibre present in the composition as sole external structurant comprises a yield stress in the range 80 Pa to 150 Pa when measured at 25 °C, and even more preferably 100 Pa to 150 Pa. The above yield stress values are important to ensure that the citrus pulp provides sufficient structuring properties to enable the detergent composition to support non-clay particles without agglomeration and without leading to phase separation of the formulation.

Detailed Description of the Invention

Detergent compositions

1. Citrus fibre Premix

[0025] Citrus fruits (normally lemons and/or limes) may be de-juiced to leave an insoluble plant cell wall material with some internally contained sugars and pectin. The 'spongy microstructure', known as albedo, may be used to make acidic, powdered citrus fibre. The structure is dried, sieved and then washed to increase the fibre content. Dried materials are typically large (with cell fragments greater than 100 microns), consisting of tightly bound/bonded fibrils). After milling a powdered citrus fibre material is obtained. This procedure leaves much of the natural cell wall intact whilst sugars are removed. The resultant swellable citrus fibre materials are typically used as food additives and are often employed for example in low fat mayonnaise.

[0026] Microscopy shows that powdered citrus fibre is a heterogeneous mixture of particles with various sizes and shapes. The majority of the material consists of aggregated lumps of cell walls and cell wall debris. However, a number of tube-like structures with an open diameter of about 10 micron, often arranged in clusters, may be identified. These, so called, xylem vessels are water transport channels that are mainly located in the peel of citrus fruits. The xylem vessels consist of stacks of dead cells, joined together to form relatively long tubes, 200 to 300 micron long. The outsides of the tubes are reinforced by lignin, which is often laid down in rings or helices, preventing the tubes from collapse due to the capillary forces acting on the tube walls during water transport.

[0027] A preferred type of powdered citrus fibre is available from Herbafoods under the trade name, Herbacel™ AQ+ type N citrus fibre. This citrus fibre has a total (soluble and insoluble) fibre content of greater than 80 wt% and soluble fibre content of greater than 20 wt%. It is supplied as a fine dried powder with low colour and has a water binding capacity of in the region of 20 kg water per kg of powder.

[0028] To obtain structure and the required yield stress in the premix, a suitable amount of powdered citrus fibre is mixed with water and subjected to high shear to open it up structurally via use of a HPH (High Pressure Homogenizer) to expose the inner microfibrils using a high shear dispersion process at low concentration, in water. It is also advantageous to include a preservative into the premix as the dispersed activated citrus fibre is biodegradable. A 2 wt% premix should be processed under high enough shear to generate the target yield stress of the premix.

[0029] It is desirable that the shear applied to the citrus fibre should not be so high as to lead to defibrillation. It has been found that increasing the homogenisation pressure beyond that previously disclosed provides further increased weight efficacy to the premix. When used, the high-pressure homogeniser is preferably operated between 50 and 1000 barg (between about 5100 kPa and about 100100 kPa). More preferably when a high-pressure homogeniser is used it is operated between 100 and 700 barg (about 10100 kPa and about 70100 kPa respectively). Most preferably the high-pressure homogeniser is operated between 300 and 500 barg (about 30100 and about 50100 kPa respectively). The more shear that is applied the less dense the resulting particles. Whilst the morphology is changed by the high shear, process aggregate size appears not to be changed and instead fibres break down and then fill the water. The shearing

process also loosens the outer parts of the fruit cell walls and these are able to form a matrix that structures the water outside the volume of the original fibre.

[0030] Whilst the premix may be left to hydrate further (or age) following high shear, it is preferred that the premix is mixed with the remaining detergent ingredients in step (b) within 72 hours, preferably within 24 hours, of step (a).

[0031] High pressure homogenised premixes are preferred over milled premixes, as the former provide a more weight effective external structurant to provide sufficient suspending duty to the detergent liquids.

[0032] Depending on how it is processed the level of citrus fibre in a premix may lie in the range of 0.05% to 6 wt%. However, it is preferred that the level of citrus fibre in the premix lies in the range of 1.5% to 3 wt%. Most preferably the level of citrus fibre in the premix lies in the range of 1.8 to 2.3 wt%. It is the yield stress of the premix used in the second stage of the process that is the main determinant for successful external structuring.

[0033] It will, however, be apparent to a skilled reader that the upper limit for concentration of citrus fibre in the premix depends on the ability of the equipment to deal with the higher viscosity, especially at higher concentrations.

[0034] The level of citrus fibre premix inclusion in the liquid detergent composition is a maximum of 0.125 wt% of citrus fibre. A preferred range for the level of citrus fibre in the liquid detergent composition is 0.05 wt % to 0.12 wt%. More preferably the level of citrus fibre premix inclusion in the liquid detergent composition is a maximum of 0.10 wt% of citrus fibre. Thus a more preferred range for the level of citrus fibre premix in the liquid detergent composition is 0.06 wt% to 0.10 wt%. Even more preferably the level of citrus fibre inclusion in the detergent composition is in the range 0.06 wt% to 0.09 wt%. Most preferably, however, the level of citrus fibre inclusion in the detergent composition is in the range 0.07 wt% to 0.08 wt%.

[0035] The amount of water in the citrus fibre premix is at least 20 times greater than the amount of citrus fibres. More preferably the amount of water in the premix is at least at least 40 times the amount of citrus fibres. The amount of water in the premix may in some instances exceed 100 times the amount of citrus fibres.

[0036] A preferred yield stress range for the citrus fibre premix is typically 80 to 250 Pa. More preferably the yield stress range for the citrus fibre premix is 80 to 180 Pa. Even more preferably, the yield stress range for the citrus fibre premix is 80 to 160 Pa. Most preferably the yield stress range for the citrus fibre premix is 80 to 150 Pa. It is advantageous that the yield stress for the citrus fibre premix is 80 Pa or more. It is also preferred that the yield stress for the citrus fibre premix is 150 Pa or less.

[0037] The citrus fibre in accordance with the present invention boosts the yield stress and the pour viscosity of the detergent composition at $21s^{-1}$ and the composition is a shear thinning liquid. It has been found that when a citrus fibre premix is used with yield stress properties outlined above, the amount of citrus fibre in the composition may be reduced whilst retaining the ability to suspend non-clay particulate material due to the maintained yield stress of the detergent liquid composition.

[0038] Citrus fibre is compatible with enzymes used in laundry and household care detergent compositions.

[0039] The citrus fibre premix may be added to the rest of the detergent liquid composition as a post dosed ingredient. Alternatively, the detergent liquid composition may be formed by starting with a citrus fibre premix followed by the addition of other ingredients as required. Some high shear is required to disperse the premix in the rest of the detergent composition fully but the duty is not as demanding as for the premix preparation.

2. Water

[0040] The detergent liquid compositions are aqueous. Hydrotropes such as propylene glycol and glycerol/glycerine may also be included as co-solvents with water. The water referred to in the composition includes both free water, which is preferably demineralised water or chlorinated demineralised water and also any 'bound' water, arising from the inclusion of components which are themselves supplied in water/demineralised water. The amount of water in the composition is at least 10 wt%. Preferably the amount of water in the composition is at least 20 wt%. Most preferably the amount of water in the composition is at least 40 wt%.

3. Detersive surfactant

[0041] The use of citrus fibre as the sole external structurant means that there are few limitations on the type or the amount of detersive surfactant in the detergent composition. However, synthetic surfactants preferably form a major part of the surfactant system. Mixtures of synthetic anionic and nonionic surfactants, or a wholly anionic mixed surfactant system or admixtures of anionic surfactants, nonionic surfactants and amphoteric or zwitterionic surfactants may all be used according to the choice of the formulator for the required cleaning duty and the required dose of the liquid detergent composition.

[0042] In addition, the surfactants forming the mixed surfactant system may be chosen from the surfactants described in 'Surface Active Agents' Vol. 1, by Schwartz & Perry, Interscience 1949, Vol. 2 by Schwartz, Perry & Berch, Interscience 1958, 'McCutcheon's Emulsifiers and Detergents' published by Manufacturing Confectioners Company or in 'Tenside

Taschenbuch', H. Stache, 2nd Edn., Carl Hauser Verlag, 1981.

[0043] The amount of detersive surfactant in the composition may be up to 70 wt%. Preferably, the amount of detersive surfactant in the composition lies in the range 4 to 60 wt%. More preferably the amount of detersive surfactant in the composition lies in the range 5 to 50 wt% or even in the range 10 to 40 wt%. Most preferably the total surfactant may be in the range 15 to 30 wt%. It will be appreciated that the optimum surfactant concentration will largely depend on the product type and the intended mode of use.

[0044] Examples of suitable synthetic anionic surfactants include: sodium lauryl sulfate, sodium lauryl ether sulfate, ammonium lauryl sulfosuccinate, ammonium lauryl sulfate, ammonium lauryl ether sulfate, sodium cocoyl isethionate, sodium lauroyl isethionate, and sodium N-lauryl sarcosinate. Preferably, the synthetic anionic surfactants comprise linear alkylbenzene sulfonate (LAS) and/or sodium alcohol ethoxy-ether sulfate (SAES), most preferably comprising high levels of sodium C12 alcohol ethoxy-ether sulfate (SLES). It is most preferred that the detergent liquid composition prepared according to the present invention comprises LAS.

[0045] Synthetic anionic surfactants may be present, for example, in amounts in the range from about 5% to about 70 wt % of the mixed surfactant system. More preferably, anionic surfactants may be present between 5% to 40 wt%. Most preferably anionic surfactants may be present between 10% to 30 wt%.

[0046] Anionic surfactant, when used, may also further include soap (that is, a salt of fatty acid). A preferred soap employed in detergent compositions according to the present invention is made by neutralisation of hydrogenated coconut fatty acid, for example Prifac® 5908 (ex Croda). Mixtures of saturated and unsaturated fatty acids may also be used.

[0047] A preferred nonionic surfactant is a C_{12}-C_{18} ethoxylated alcohol, comprising 3 to 9 ethylene oxide units per molecule. More preferred are C_{12}-C_{15} primary, linear ethoxylated alcohols with on average between 5 and 9 ethylene oxide groups. Most preferably, linear ethoxylated alcohols with an average of 7 ethylene oxide groups are employed.

[0048] A preferred detersive surfactant system comprises synthetic anionic with nonionic detergent active materials and optionally amphoteric surfactant, including amine oxide.

[0049] Another preferred detersive surfactant system comprises two different anionic surfactants, preferably linear alkyl benzene sulphonate and a sulfate, for example LAS and SLES.

[0050] The detergent compositions may further comprise an amphoteric surfactant, wherein the amphoteric surfactant is present in a concentration of 1 to 20 wt%. Preferably the detergent compositions comprise an amphoteric surfactant present in a concentration of 2 to 15 wt%. More preferably the detergent compositions comprise an amphoteric surfactant present in a concentration of 3 to 12 wt% of the mixed surfactant system. Typical examples of suitable amphoteric and zwitterionic surfactants include: alkyl betaines, alkylamido betaines, amine oxides, aminopropionates, aminoglycinates, amphoteric imidazolinium compounds, alkyldimethylbetaines or alkyldipolyethoxybetaines.

4. Suspended non-clay particles

[0051] The composition prepared in accordance with the present invention comprises suspended non-clay particles. These particles are preferably solid; that is to say they are neither liquid nor gas. However, within the term solid we include particles with either rigid or deformable solid shells which may then contain fluids. For example the solid particles may be microcapsules such as perfume encapsulates (encaps), or care additives in encapsulated form. The particles may take the form of insoluble ingredients such as silicones, quaternary ammonium materials, insoluble polymers, insoluble optical brighteners and other known benefit agents as described, for example, in EP1328616. The amount of suspended particles in the liquid detergent composition may be from 0.001 to up to 2 wt%. The amount of suspended particles in the liquid detergent may also be up to 5 wt% or even 10 wt%.

[0052] The average particle diameter of the microcapsules lies in the range from 1 to 100 micrometer and at least 90 wt% of the microcapsules preferably possess a diameter in this range. More preferably, 90 wt% of the microcapsules have a diameter in the range 2 to 50 micrometers, even more preferably 5 to 50 micrometers. Most preferred are microcapsules with diameters less than 30 micrometers.

[0053] It is advantageous to have a very narrow particle size distribution, for instance 90 wt% of microcapsules in the range 8 to 11 microns. Microcapsules in the range 2 to 5 microns cannot be dispersed so effectively due to the high surface area of the smaller particles.

[0054] Preferably the composition comprises at least 0.01 wt% of microcapsules, preferably with an anionic charge. Such microcapsules may deliver a variety of benefit agents by deposition onto substrates such as laundry fabric. To obtain maximum benefit they should be well dispersed through the liquid detergent composition and the vast majority of the microcapsules must not be significantly agglomerated. Any microcapsules that become agglomerated during manufacture of the liquid remain so in the container and will thus be dispensed unevenly during use of the composition. This is highly undesirable. The contents of the microcapsules are normally liquid. For example, fragrances, oils, fabric softening additives and fabric care additives are possible contents. Preferred microcapsules are particles termed core-in-shell microcapsules. As used herein, the term core-in-shell microcapsules refers to encapsulates whereby a shell which is substantially or totally water-insoluble at 40°C surrounds a core which comprises or consists of a benefit agent

(which is either liquid or dispersed in a liquid carrier).

[0055] Suitable microcapsules are those described in US 5066419 which have a friable coating, preferably an aminoplast polymer. Preferably, the coating is the reaction product of an amine selected from urea and melamine, or mixtures thereof, and an aldehyde selected from formaldehyde, acetaldehyde, glutaraldehyde or mixtures thereof. Preferably, the coating is from 1 to 30 wt% of the particles. Other suitable shell material may be selected from but not limited to: (poly)urea, (poly)urethane, starch/polysaccharide, xyloglucan and aminoplasts.

[0056] Core-in-shell microcapsules of other kinds are also suitable for use in the present invention. Ways of making such other microcapsules benefit agents such as perfume include precipitation and deposition of polymers at the interface such as in coacervates, as disclosed in GB 751600, US 3341466 and EP 385534, as well as other polymerisation routes such as interfacial condensation, as described in US 3577515, US2003/0125222, US 6020066 and WO 03/101606.

[0057] Microcapsules having polyurea walls are disclosed in US 6797670 and US 6586107. Other patent applications specifically relating to use of melamine-formaldehyde core-in-shell microcapsules in aqueous liquids are WO 98/28396, WO 02/074430, EP 1244 768, US2004/0071746 and US2004/0142868.

[0058] Perfume encapsulates are a preferred type of microcapsule suitable for use in the composition of the present invention.

[0059] A preferred class of core-in-shell perfume microcapsule comprises those disclosed in WO 2006/066654. These comprise a core having from about 5% to about 50 wt% of perfume dispersed in from about 95% to about 50 wt% of a carrier material. This carrier material preferably is a non-polymeric solid fatty alcohol or fatty ester carrier material, or mixtures thereof. Preferably, the esters or alcohols have a molecular weight of from about 100 to about 500 and a melting point from about 37 °C to about 80°C, and are substantially water-insoluble. The core comprising the perfume and the carrier material are coated in a substantially water-insoluble coating on their outer surfaces. Similar microcapsules are disclosed in US 5154842 and these are also suitable.

[0060] The microcapsules may attach to suitable substrates, for example to provide persistent fragrance that is desirably released after the cleaning process is complete.

[0061] In order to deliver microcapsules comprising fragrance, often referred to as fragrance encapsulates (or "encaps") to fabric, the microcapsules must be added to a fabric washing liquid. Since these microcapsules are less dense than an isotropic liquid phase, the microcapsules cannot be suspended in a Newtonian (non-structured fabric washing liquid as the microcapsules "cream" or float to the surface if placed in the fabric washing liquid.

[0062] One type of solid particle to be suspended is a visual cue, for example the type of flat film cue described in EP 13119706. The cue may itself contain a segregated component of the detergent composition. However, because the cue must be water-soluble, yet insoluble in the composition, it is conveniently made from a modified polyvinyl alcohol that is insoluble in the presence of the mixed surfactant system. In which case, the detergent composition preferably comprises anionic surfactant at a level of at least 5 wt%.

[0063] Perfume encapsulates are a preferred type of microcapsule suitable for use as non clay particles in the present invention.

[0064] Deposition aids may also be present located on the surface of the microcapsules. These may advantageously be selected from non-ionic materials, preferably cellulose derivatives and polyesters and provide better substantivity to a plurality of substrates. Particularly preferred polysaccharide additional deposition aids include dextran, hydroxy-propyl methyl cellulose, hydroxy-ethyl methyl cellulose, hydroxy-propyl guar, hydroxy-ethyl ethyl cellulose, methyl cellulose, locust bean gum, xyloglucan, guar gum. Particularly preferred polyester additional deposition aids include polymers having one or more nonionic hydrophilic components comprising oxyethylene, polyoxyethylene, oxypropylene or polyoxypropylene segments, and, one or more hydrophobic components comprising terephthalate segments.

[0065] The microcapsules may attach to suitable substrates, for example, to provide persistent fragrance that is desirably released after the cleaning process is complete.

5. Liquid detergent compositions

[0066] In accordance with the present invention the detergent compositions have sufficient initial yield stress, also called critical stress, in the range 0.3 Pa to 1.2 Pa, to suspend the particles and to be stable. More preferably the initial yield stress of the composition in accordance with the present invention is in the range 0.3 Pa to 1.0 Pa or 0.35 Pa to 0.8 Pa. Even more preferably the initial yield stress of the composition in accordance with the present invention is in the range 0.4 Pa to 0.75 Pa. All yield stress values measured at 25°C. These levels of yield stress have been found capable of suspending particles of markedly different density from the bulk liquid. A yield stress at the lower end of this range has been found sufficient to suspend most types of perfume encapsulates.

[0067] The sole structurant citrus fibre external structuring system used in the compositions of the present invention stays dispersed; neither floating (to give bottom clear layer separation) nor sinking (to give top clear layer separation). This self suspension is achieved by ensuring that the structuring system wants to occupy all the volume of the detergent liquid. This is a function of the amounts of citrus fibre used, the yield stress of the premix and its processing.

[0068] The detergent liquid may be formulated as a concentrated detergent liquid for direct application to a substrate, or for application to a substrate following dilution, such as dilution before or during use of the liquid composition by the consumer or in washing apparatus.

[0069] Cleaning may be carried out by simply leaving the substrate in contact for a sufficient period of time with a liquid medium constituted by or prepared from the liquid cleaning composition. Preferably, however, the cleaning medium on or containing the substrate is agitated.

6. Product Form

[0070] The liquid detergent compositions according to the present invention are preferably concentrated liquid cleaning compositions. The liquid compositions have a physical form, which ranges from a pourable liquid to a pourable gel. These forms are conveniently characterised by the product viscosity. In these definitions, and unless indicated explicitly to the contrary, throughout this specification, all stated viscosities are those measured at a shear rate of 21 s^{-1} and at a temperature of 25°C. This shear rate is the shear rate that is usually exerted on the liquid when poured from a bottle. The liquid detergent compositions made according to the invention are shear-thinning liquids.

[0071] Pourable liquid detergent compositions preferably have a maximum viscosity of 2000 m Pa.s quoted at 21 s^{-1}. More preferably liquid detergent compositions have a viscosity of not more than 1500m Pa.s. Still more preferably, liquid detergent compositions have a viscosity of not more than 1000 m Pa.s.

[0072] Liquid detergent compositions which are pourable gels preferably have a viscosity of at least 1,000 mPa.s but no more than 6,000 mPa.s. More preferably liquid detergent compositions which are pourable gels, have a viscosity of no more than 4,000 mPa.s. Still more preferably liquid detergent compositions which are pourable gels have a viscosity of no more than 3,000 mPa.s and especially no more than 2,000 mPa.s.

[0073] For the purpose of the invention a composition is considered physically stable when it remains homogeneous with dispersed and suspended perfume encapsulates over a period of 3 months at temperatures from 5 to 25 °C and over a period of 2 months at temperatures at 37°C.

[0074] By concentrated, is meant low dosage levels of detergent applied to the wash. Typical dosages for concentrated liquids are between 30 and 45 ml, more preferably 35 to 40 ml, most preferably, 35 ml. Surfactant concentrations may typically range from: 10 to 60 wt% total surfactant; more preferably 15 to 40 wt% total surfactant; most preferably between 20 wt% and 30 wt% total surfactant.

[0075] The liquid detergent compositions are preferably concentrated liquid cleaning compositions. The liquid compositions are also preferably pourable liquids.

[0076] Throughout this specification, all stated viscosities are those measured at a shear rate of 21s^{-1} and at a temperature of 25°C, unless stated to be otherwise. This shear rate is the shear rate that is usually exerted on the liquid when poured from a bottle. The liquid detergent compositions according to the invention are shear-thinning liquids.

7. Manufacturing process

[0077] Structured detergent compositions are preferably prepared starting with the sheared citrus fibre and water premix and then adding the other detergent ingredients in their normal order of addition. This has the further advantage that dispersion of the citrus fibre by high shear continues during the addition of the later ingredients rather than as a post shearing step, thereby reducing the batch time. The liquid may be de-aerated before filling it into containers. However, the external structuring system allows for more process flexibility and this step is not essential.

8. Optional ingredients

[0078] Citrus fibre has been found to be compatible with usual ingredients that may be found in detergent liquids. Among which there may be mentioned, by way of example: polymeric thickeners; enzymes, particularly: lipase, cellulase, protease, mannanase, amylase and pectate lyase; cleaning polymers, including ethoxylated polyethylene imines (EPEI) and polyester soil release polymers; chelating agents or sequestrants, including HEDP (1-Hydroxyethylidene -1,1,-di-phosphonic acid) which is available, for example, as Dequest® 2010 from Thermphos; detergency builders; hydrotropes; neutralising and pH adjusting agents; optical brighteners; antioxidants and other preservatives, including Proxel®; other active ingredients, processing aids, dyes or pigments, carriers, fragrances, suds suppressors or suds boosters, chelating agents, clay soil removal/ anti-redeposition agents, fabric softeners, dye transfer inhibition agents, and transition metal catalyst in a composition substantially devoid of peroxygen species.

[0079] These and further possible ingredients for inclusion are further described in WO 2009/153184.

Packaging

[0080] The compositions of the present invention may be packaged in any form of container. Typically a plastic bottle with a detachable closure/pouring spout may be used. The bottle may be rigid or deformable. When a deformable bottle is used, this allows the bottle to be squeezed to aid dispensing of the composition. If clear bottles are used they may be formed from polyethylene terephthalate (PET). Polyethylene or clarified polypropylene may also be used to form the container. Preferably the container or bottle is clear enough that the liquid composition with any visual cues therein, is visible when viewed from the outside of the container. The container may be provided with one or more labels. In addition, the container may be provided with a shrink wrap sleeve which is desirably at least partially transparent. For example, 50% of the area of the sleeve may be transparent. The adhesive used to secure any transparent label should preferably not adversely affect the transparency of the container.

EXAMPLES

[0081] The invention will now be further described with reference to the following non-limiting examples.

Abbreviations

[0082] The following abbreviated names used in these examples have the following meanings:

Water	is Demineralised water.
Glycerol	is hydrotrope.
MPG	is Monopropylene Glycol (hydrotrope).
Neodol NI	is a nonionic surfactant ex Shell
NaOH	is 50% sodium hydroxide base.
LAS acid	is linear alkyl benzene sulphonic acid anionic surfactant.
TEA	is Triethanolamine base not present.
SLES (3EO)	is SLES 3EO anionic surfactant.
Dequest 2066	is Diethylenetriamine penta(methylene phosphonic acid (or Heptasodium DTPMP) sequestrant ex Thermphos.
HPH	High pressure homogenizer.
Encapsulated fragrance	is encapsulated perfume, Oasis Cap Det B72 ex Givaudan.
Preservative	is Proxel GXL™ antimicrobial preservative, 20% solution of 1,2 benzisothiazolin-3-one in dipropylene glycol and water ex Arch Chemicals.
Perfume	is free oil perfume.
Tinopal 5BM-GX	is a fluorescer.

Experimental

General procedure - preparation of citrus fibre premix.

Procedure 1

[0083] 2 kg of 2 wt% citrus fibre premix was prepared using citrus fibre powder (Herbacel AQ plus N Citrus Fibre (ex: Herbafoods)) as set out in Table 1 .

Table 1

Material	% As supplied	Weight (g)
26°FH water	97.92	1958.4
Proxel GXL™	0.08	1.6
Herbacel AQ+ type N	2	40

[0084] The citrus fibre powder was added slowly to a mixture of 26°FH water and preservative, Proxel GXL™, and the reactants stirred with an agitator with overhead drive operated at 200 rpm for 15 minutes ensuring that clumping did not occur. This allowed the citrus fibre to hydrate sufficiently prior to activation. The citrus fibre powder/ water premix

was then processed using an APV2000 laboratory high pressure homogenizer available from SPX operating at a pressure of 500 barg (approximately 50100 kPa).

2. Procedure 2

[0085] 2 kg of a second citrus fibre premix was prepared using citrus fibre powder (Herbacel AQ plus N Citrus Fibre (ex: Herbafoods)), in the amounts set out in Table 1. The citrus fibre was added slowly to a mixture of de-mineralised water and preservative, Proxel GXL™, and the reactants stirred with an agitator with overhead drive operated at 200 rpm for 15 minutes ensuring that clumping did not occur. This allowed the citrus fibre to hydrate sufficiently prior to shear activation. The citrus fibre powder/demineralised water premix was then homogenized at a pressure of 500 barg (approximately 50100 kPa) using an APV2000 laboratory high pressure homogenizer available from SPX. The citrus fibres were then used to prepare the detergent compositions within 72 hours.

Experiment 1 - Preparation of detergent compositions using activated citrus fibre at a concentration of less than 0.1 wt% using citrus fibre prepared using procedure 1 or procedure 2

[0086] A series of detergent compositions with various levels of citrus fibre were prepared in a 5 litre capacity vessel by adding the components listed in Table 2 in the order set out therein. The compositions were prepared using a single pass protocol, which involved pumping the composition, through a mill (typically a Silverson L5M or L5T) using a total energy of between 1.2 kJ/kg and 5 kJ/kg. In Table 2, 'as 100%' defines the amount of reagent required in the final composition based on 100% reagent supplied.

Table 2 - Summary of detergent liquid compositions prepared using activated citrus fibre at a concentration of less than 0.1 wt%

Composition	Example 1a 0.06 % citrus fibre prepared using procedure 2.	Example 1b 0.07 % citrus fibre prepared using procedure 2.	Example 1c 0.08 % citrus fibre prepared using procedure 2.	Example 1d 0.09 % citrus fibre prepared using procedure 2.	Example 1e 0.06 % citrus fibre prepared using procedure 1.
Reagent	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient
Water	59.27	59.26	59.25	59.24	
26 °FH water	-	-	-	-	59.27
Encapsulated Fragrance	0.40	0.40	0.40	0,40	0.40
Citrus Fibre (from 2% premix)	0.06	0.07	0.08	0.09	0.06
Tinopal 5BM-GX	0.08	0.08	0.08	0.08	0.08
MPG	2.00	2.00	2.00	2.00	2.00
Glycerol	5.00	5.00	5.00	5.00	5.00
NaOH	1.20	1.20	1.20	1.20	1.20
TEA	1.69	1.69	1.69	1.69	1.69
Citric Acid	0.00	0.00	0.00	0.00	0.00
Neodol NI	13.72	13.72	13.72	13.72	13.72
LAS acid	9.15	9.15	9.15	9.15	9.15
Saturatedfatty acid	1.50	1.50	1.50	1.50	1.50
SLES (3EO)	4.57	4.57	4.57	4.57	4.57
Dequest 2066	0.34	0.34	0.34	0.34	0.34

(continued)

Composition	Example 1a 0.06 % citrus fibre prepared using procedure 2.	Example 1b 0.07 % citrus fibre prepared using procedure 2.	Example 1c 0.08 % citrus fibre prepared using procedure 2.	Example 1d 0.09 % citrus fibre prepared using procedure 2.	Example 1e 0.06 % citrus fibre prepared using procedure 1.
Reagent	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient
Base liquid total	98.98	98.98	98.98	98.98	98.98
Preservative	0.02	0.02	0.02	0.02	0.02
Perfume	1.00	1.00	1.00	1.00	1.00
Total	100%	100%	100%	100%	100%

[0087] The yield stress values of the citrus fibre premix used in the compositions detailed in Table 2 and the yield stress values of the prepared compositions from Table 2 were measured along with the viscosity of the compositions. The recorded values are provided in Table 3 below. The compositions were studied and the stability assessed over a 12 week period using an aeration test and a 'dip test' as described below. The results of the assessment are provided in Table 3.

Aeration Test

[0088] The liquid detergent compositions were monitored for the appearance of air bubbles in the liquid compositions. The presence of air bubbles in the composition may result in the break-down of the structure of the detergent composition as a result of collapse of the citrus fibre. The presence of air bubbles in the detergent compositions was graded from A0 to A3. A value of A0 indicated zero or minimal air bubbles and hence a pass whilst a value of A2 to A3 indicated the presence of an extensive amount of air bubbles. The detergent compositions prepared according to the present invention all passed the aeration test.

Photographic stability tests or 'dip' tests

'Dip test' - test for 'drop' or 'lift' visible inside detergent composition

[0089] The liquid detergent compositions were also monitored for the appearance of a non-uniform region developing at the top or the bottom of the composition. The detergent liquid compositions were visually assessed for the presence of a non-uniform region discernible towards the top or bottom of the detergent liquid composition which is indicative of a break-down in the uniformity of the detergent liquid and is different in appearance to the remainder of the detergent liquid. A drop or dip of 1 to 2mm when the liquid composition is held at room temperature or 5 °C is acceptable and therefore a pass. A drop of greater than or equal to 5 mm when the liquid composition is held at room temperature (25°C) or 5°C is deemed a product failure. The detergent compositions prepared according to the present invention all passed the 'dip-test'.

Table 3 - Summary of stability tests for detergent liquid compositions prepared using citrus fibre premix to give a concentration of less than 0.1 wt% in formulation using procedure 1 or 2

	Citrus fibres (wt% in comp)	Yield Stress of citrus fibre premix (Pa)	Specific Energy Input (kJ/kg)	Viscosity at $21s^{-1}$ mPa.s	Initial Yield Stress of comp. (Pa)	Stability test at 5 ° C	Stability test at room temperature	Stability test at 37 ° C	Overall Stability test at 12 weeks
1a	0.06	150	4.4	619	0.32	Pass	Pass	Pass	Pass
1b	0.07	150	4.4	641	0.42	Pass	Pass	Pass	Pass
1c	0.08	150	4.4	650	0.55	Pass	Pass	Pass	Pass
1d	0.09	150	4.4	681	0.61	Pass	Pass	Pass	Pass
1e	0.06	70	2.9	480	0.19	FAIL	FAIL	FAIL	FAIL

[0090] It can be seen from Table 3 that stable compositions were obtained over a 12 week assessment period for compositions with citrus fibre levels below 0.1 wt% based on using an initial premix concentration of 2 wt%, when the yield stress of the citrus fibre premix was 80 Pa or above, leading to compositions with an initial yield stress of between 0.3 Pa and 1.0 Pa. However, for a composition in which the yield stress of the citrus fibre premix was below 80 Pa, leading to a composition with an initial yield stress of less than 0.3 Pa, the formulation failed the stability and storage test.

[0091] The specific energy input (kJ/kg) defines the mixing conditions which impart sufficient energy to give a consistent structure throughout the detergent liquid. This energy may be attained by varying the mixing power and the resonance time within the mixer. A specific energy input of between 1.2 and 5 kJ/kg provides consistent citrus fibre structurant dispersal.

Experiment 2. Preparation of detergent compositions with citrus fibre at a concentration of 0.125 wt% or less using citrus fibre premix prepared using procedure 2 and a specific energy of 1.2 kJ/kg or less

[0092] A series of detergent compositions with varying levels of citrus fibre were again prepared by adding the components listed in Table 4 in the order set out therein, in a 5 litre capacity vessel. The compositions were prepared using a single pass protocol, which involves pumping the composition, through a mill (typically a Silverson L5M or L5T) at a total energy of 1.2 kJ/kg or less. In Table 4, 'as 100%' defines the amount of reagent required in the final composition based on 100% reagent supplied.

Table 4 - Range of detergent compositions with citrus fibre at a concentration of 0.125 wt% or less using citrus fibre premix prepared using procedure 2 and a specific energy of 1.2kJ/kg or less

Composition	**Example 2a 0.075 % citrus fibre**	**Example 2b 0.10 % citrus fibre**	**Example 1c 0.125 % citrus fibre**
Reagent	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient
Water	59.26	59.23	59.21
Encapsulated Fragrance	0.40	0.40	0.40
Citrus Fibre (from 2% premix)	0.075	0.10	0.125
Tinopal 5BM-GX	0.08	0.08	0.08
MPG	2.00	2.00	2.00
Glycerol	5.00	5.00	5.00

(continued)

Composition	Example 2a 0.075 % citrus fibre	Example 2b 0.10 % citrus fibre	Example 1c 0.125 % citrus fibre
Reagent	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient
NaOH	1.20	1.20	1.20
TEA	1.69	1.69	1.69
Citric Acid	0.00	0.00	0.00
Neodol NI	13.72	13.72	13.72
LAS acid	9.15	9.15	9.15
Saturated fatty acid	1.50	1.50	1.50
SLES (3EO)	4.57	4.57	4.57
Dequest 2066	0.34	0.34	0.34
Base liquid total	98.98	98.98	98.98
Preservative	0.02	0.02	0.02
Perfume	1.00	1.00	1.00
Total	100%	100%	100%

[0093] The yield stress values of the citrus fibre concentrations used in the compositions detailed in Table 4 and the yield stress values of the prepared compositions from Table 4 were measured along with the viscosity of the compositions. The recorded values are provided in Table 5 below. The compositions were studied and the stability again assessed over a 12 week period, and these values are also provided in Table 5.

Table 5 - Summary of detergent liquid compositions with citrus fibre at a concentration of 0.125 wt% or less using citrus fibre premix prepared using procedure 2 and a specific energy of 1.2 kJ/kg or less

Citrus fibres (wt% in comp.)	Yield Stress of citrus fibre (Pa)	Specific Energy (kJ/kg)	Viscosity at $21s^{-1}$ mPa.s	Initial Yield Stress of comp. (Pa)	Stability test at 5 ° C	Stability test at room temperature	Stability test at 37 ° C	Overall Stability test at 12 weeks
0.075	141	1.2	690	0.45	Pass	Pass	Pass	Pass
0.10	141	1.2	650	0.62	Pass	Pass	Pass	Pass
0.10	89	1.1	814	0.55	Pass	Pass	Pass	Pass
0.125	140	1.2	768	0.73	Pass	Pass	Pass	Pass

[0094] It can be seen from Table 5 that stable compositions were obtained over a 12 week assessment period for compositions with citrus fibre levels at or below 0.125 wt% based on an initial premix concentration of 2 wt%, when the yield stress of the citrus fibre is 80 Pa or more, leading to compositions with an initial yield stress of between 0.3 Pa and 1.0 Pa even for compositions prepared with lower specific energies than the compositions given in Table 3.

Experiment 3. Preparation of detergent compositions with citrus fibre at a concentration of 0.125 wt% or less using citrus fibre premix prepared using procedure 2

[0095] A series of detergent compositions with varying levels of citrus fibre were prepared by adding the components

listed in Table 2 in the order set out therein, in a 5 litre capacity vessel. The compositions were prepared using a single pass protocol, which involves pumping the composition through a mill (typically a Silverson L5M or L5T) at a total energy of a range between 1.2 KJ/Kg and 5 KJ/kg.

[0096] In Table 6, 'as 100%' defines the amount of reagent required in the final composition based on 100% reagent supplied. The compositions in Table 6 contain citric acid, have increased amounts of glycerol, but contain no monopropylene glycol (MPG).

Table 6 - Lower viscosity detergent compositions with increased glycerol, no MPG and 0.62% citric acid with citrus fibre at a concentration of 0.125 wt% or less using citrus fibre premix prepared using procedure 2

Composition material	**Example 3a 0.10 % citrus fibre**	**Example 3b 0.10 % citrus fibre**	**Example 3c 0.125 % citrus fibre**
Reagent	As 100% Active ingredient	As 100% Active ingredient	As 100% Active ingredient
Water	58.61	58.61	58.585
Encapsulated Fragrance	0.40	0.40	0.40
Citrus Fibre (from 2% premix)	0.10	0.10	0.125
Tinopal 5BM-GX	0.08	0.08	0.08
MPG	0.00	0.00	0.00
Glycerol	7.00	7.00	7.00
NaOH	1.20	1.20	1.20
TEA	1.69	1.69	1.69
Citric Acid	0.62	0.62	0.62
Neodol NI	13.72	13.72	13.72
LAS acid	9.15	9.15	9.15
Saturated fatty acid	1.50	1.50	1.50
SLES (3EO)	4.57	4.57	4.57
Dequest 2066	0.34	0.34	0.34
Base liquid total	98.98	98.98	98.98
Preservative	0.02	0.02	0.02
Perfume	1.00	1.00	1.00
Total	100%	1 00%	100%

[0097] The yield stress values of the citrus fibre concentrations used in the compositions detailed in Table 6 and the yield stress values of the prepared compositions from Table 6 were measured along with the viscosity of the compositions. The recorded values are provided in Table 7 below. The compositions were studied and the stability assessed over a 12 week period, and these values are also provided in Table 7.

Table 7 - Summary of detergent liquid compositions with citrus fibre at a concentration of 0.125 wt% or less using citrus fibre premix prepared using procedure 2

	Citrus fibres (wt% in comp.)	Yield Stress of citrus fibre (Pa)	Specific Energy (kJ/kg)	Initial Yield Stress of comp. (Pa)	Stability test at 5 ° C	Stability test at room temperature	Stability test at 37 ° C	Overall Stability test at 12 weeks
3a	0.10	141	1.2	0.41	Pass	Pass	Pass	Pass
3b	0.10	128	2.8	0.54	Pass	Pass	Pass	Pass
3c	0.125	128	2.8	0.77	Pass	Pass	Pass	Pass

[0098] It can be seen from Table 7 that stable compositions with 1.2 to 5 kJ/kg energy were obtained over a 12 week assessment period for compositions with citrus fibre levels at or below 0.125% based on an initial premix concentration of 2 wt% and prepared using procedure 2, when the yield stress of the citrus fibre premix is 80 Pa or more, leading to compositions with an initial yield stress of between 0.4 Pa and 1.0 Pa, even for compositions with reduced viscosity.

[0099] Therefore, From these experiments it can be concluded that by using sufficient shear (pressure) to get the yield stress of a citrus fibre premix in a high range it is possible to reduce the amount of citrus fibre used as sole external structurant in detergent liquids whilst retaining the support framework provided by the citrus fibre.

[0100] That is, it is possible to reduce the level of citrus fibre in externally structured detergent liquids if a citrus fibre premix is prepared and used with a yield stress of at least 80 Pa. Such a citrus fibre premix is obtained by subjecting the citrus fibres to a minimum level of energy and a minimum pressure during a high pressure homogenisation (HPH) step. That is, the yield stress value of the citrus fibre premix is related to the yield stress values of the externally structured detergent liquids, and the required low levels of citrus fibre in the externally structured detergent liquids is obtainable by use of a citrus fibre premix with a minimum yield stress of 80 Pa to provide a detergent composition with a yield stress of at least 0.3 Pa.

[0101] Using a citrus fibre premix with a yield stress values of less than 80 Pa as the sole structurant resulted in the detergent formulations failing the stability tests. That is, failed structurant systems either sediment or show a breakdown in the structure of the formulation by the development of a substantially clear upper layer in the formulation.

[0102] Therefore the yield stress of the CP premix and concentration inclusion level of the citrus fibres in the detergent are important factors in preparing stable structured detergent formulations which are ultimately stable.

Claims

1. A process to manufacture an externally structured aqueous liquid detergent composition having a yield stress in the range of 0.3 Pa to 1.2 Pa, the composition comprising at least 10 wt% water; at least 3 wt% detersive surfactant; at least 0.01 wt% non-clay particles; and consisting essentially of 0.05 to 0.125 wt% citrus fibre as sole external structurant comprising the steps of:

   a) subjecting citrus fibre to mechanical processing comprising application of shear in the presence of 20 to 120 times the amount of water based on the citrus fibres, the shear being sufficient to cause structural disruption and hydration of the citrus fibres to form a structuring premix comprising dispersed pulped citrus fibre and having a yield stress in the range of 80 to 250 Pa, preferably 80 to 180 Pa;

   b) combining and further dispersing the structuring premix formed in step (a) and having a yield stress in the range of 80 to 250 Pa, preferably 80 to 180 Pa with detergent liquid components in the absence of any other external structurants to form the aqueous externally structured liquid detergent composition with a yield stress in the range of 0.3 to 1.2 Pa when measured at 25 °C.

2. A process according to claim 1 wherein the structuring premix formed in step (a) has a yield stress in the range 80 Pa to 160 Pa, preferably 80 Pa to 150 Pa, and even more preferably 100 Pa to 150 Pa, when measured at 25 °C

3. A process according to any preceding claim wherein the structuring premix formed in step (a) comprises from 1.5

to 3 wt% citrus fibre, preferably about 2 wt%.

4. A process according to any preceding claim wherein the detergent composition formed is a laundry detergent composition.

5. A process according to any preceding claim wherein the composition comprises at least 0.1 wt% non-clay particles, preferably at least 0.2 wt% non-clay particles.

6. A process according to any preceding claim wherein the non-clay particles are be selected from the group comprising: visual cues, perfume encapsulates, care additives and cleaning ingredients.

7. A process according to any preceding claim wherein the non-clay particles are perfume encapsulates.

8. An externally structured aqueous isotropic liquid detergent composition having a yield stress in the range of 0.3 Pa to 1.2 Pa at 25°C, comprising:

i) at least 10 wt% water;
ii) at least 3 wt% detersive surfactant;
iii) at least 0.01 wt% non-clay particles; and
iv) an external structurant,

characterised in that the external structurant consists essentially of from 0.05 to 0.125 wt% citrus fibre and the composition is obtainable by the process according to any preceding claim.

9. A detergent composition obtainable according to claim 8 wherein the yield stress of the detergent composition is in the range of from 0.30 Pa to 1.0 Pa, preferably in the range of from 0.35 Pa to 0.85 Pa when measured at 25 °C.

10. A detergent composition obtainable according to claim 8 or 9 wherein the citrus fibre is present in the detergent composition in a range of from 0.06 wt% to 0.10 wt%, preferably from 0.06 wt% to 0.09 wt%, and more preferably from 0.07 wt% to 0.08 wt%.

Patentansprüche

1. Verfahren zur Herstellung einer extern strukturierten, wässrigen flüssigen Reinigungsmittelzusammensetzung mit einer Fließspannung in dem Bereich von 0,3 Pa bis 1,2 Pa, wobei die Zusammensetzung mindestens 10 Gew.-% Wasser, mindestens 3 Gew.-% Reinigungstensid, mindestens 0,01 Gew.-% Nicht-Ton-Partikel umfasst und im Wesentlichen aus 0,05 bis 0,125 Gew.-% Citrusfaser als alleiniges externes Strukturierungsmittel besteht, umfassend die Schritte:

a) die Citrusfaser wird einer mechanischen Behandlung unterworfen, umfassend die Anwendung von Scherung in Gegenwart der 20- bis 120-fachen Menge Wasser, bezogen auf die Citrusfasern, wobei die Scherung ausreichend ist, um die strukturelle Zerstörung und die Hydratation der Citrusfasern zu bewirken, um eine strukturierende Vormischung zu bilden, die eine dispergierte Citrusfaser-Pulpe umfasst und eine Fließspannung in dem Bereich von 80 bis 250 Pa, vorzugsweise 80 bis 180 Pa, aufweist,
b) Kombinieren und weiteres Dispergieren der strukturierenden Vormischung, die in Schritt (a) gebildet wird und eine Fließspannung in dem Bereich von 80 bis 250 Pa, vorzugsweise von 80 bis 180 Pa, aufweist, mit flüssigen Reinigungsmittelbestandteilen in Abwesenheit irgendwelcher anderer externer Strukturierungsmittel, um die extern strukturierte, wässrige flüssige Reinigungsmittelzusammensetzung mit einer Fließspannung in dem Bereich von 0,3 bis 1,2 Pa, wenn bei 25°C gemessen wird, zu bilden.

2. Verfahren nach Anspruch 1, wobei die in Schritt (a) gebildete strukturierende Vormischung eine Fließspannung in dem Bereich von 80 Pa bis 160 Pa, vorzugsweise von 80 Pa bis 150 Pa und sogar bevorzugter von 100 Pa bis 150 Pa, wenn bei 25°C gemessen wird, aufweist.

3. Verfahren nach irgendeinem vorhergehenden Anspruch, wobei die in Schritt (a) gebildete strukturierende Vormischung 1,5 bis 3 Gew.-% Citrusfaser, vorzugsweise etwa 2 Gew.-%, umfasst.

4. Verfahren nach irgendeinem vorhergehenden Anspruch, wobei die gebildete Reinigungsmittelzusammensetzung eine Waschmittelzusammensetzung ist.

5. Verfahren nach irgendeinem vorhergehenden Anspruch, wobei die Zusammensetzung mindestens 0,1 Gew.-% Nicht-Ton-Partikel, vorzugsweise mindestens 0,2 Gew.-% Nicht-Ton-Partikel, umfasst.

6. Verfahren nach irgendeinem vorhergehenden Anspruch, wobei die Nicht-Ton-Partikel aus der Gruppe ausgewählt sind, die umfasst: visuelle Hinweise, Parfümeinkapselungen, Pflegeadditive und Reinigungsbestandteile.

7. Verfahren nach irgendeinem vorhergehenden Anspruch, wobei die Nicht-Ton-Partikel Parfüm-Einkapselungen sind.

8. Extern strukturierte, isotrope wässrige flüssige Reinigungsmittelzusammensetzung mit einer Fließspannung in dem Bereich von 0,3 Pa bis 1,2 Pa bei 25°C, umfassend:

   i) mindestens 10 Gew.-% Wasser,
   ii) mindestens 3 Gew.-% Reinigungstensid,
   iii) mindestens 0,01 Gew.-% Nicht-Ton-Partikel und
   iv) ein externes Strukturierungsmittel,

   dadurch gekennzeichnet, dass das externe Strukturierungsmittel im Wesentlichen aus 0,05 bis 0,125 Gew.-% Citrusfaser besteht und die Zusammensetzung durch das Verfahren nach irgendeinem vorhergehenden Anspruch erhältlich ist.

9. Reinigungsmittelzusammensetzung, erhältlich nach Anspruch 8, wobei die Fließspannung der Reinigungsmittelzusammensetzung in dem Bereich von 0,30 Pa bis 1,0 Pa, vorzugsweise in dem Bereich von 0,35 Pa bis 0,85 Pa, wenn bei 25°C gemessen wird, liegt.

10. Reinigungsmittelzusammensetzung, erhältlich nach Anspruch 8 oder 9, wobei die Citrusfaser in der Reinigungsmittelzusammensetzung in einem Bereich von 0,06 Gew.-% bis 0,10 Gew.-%, vorzugsweise von 0,06 Gew.-% bis 0,09 Gew.-% und bevorzugter von 0,07 Gew.-% bis 0,08 Gew.-%, vorliegt.

Revendications

1. Procédé de fabrication d'une composition de détergent liquide aqueuse structurée en externe ayant une contrainte d'écoulement dans l'intervalle de 0,3 Pa à 1,2 Pa, la composition comprenant au moins 10 % en masse d'eau ; au moins 3 % en masse de tensioactif détersif ; au moins 0,01 % en masse de particule de non-argile ; et consistant essentiellement en de 0,05 à 0,125 % en masse de fibre d'agrume comme seul structurant externe comprenant les étapes consistant :

   a) à soumettre une fibre d'agrume à un traitement mécanique comprenant l'application de cisaillement en présence de 20 à 120 fois la quantité d'eau sur la base des fibres d'agrumes, le cisaillement étant suffisant pour occasionner une rupture structurelle et une hydratation des fibres d'agrumes pour former un pré-mélange structurant comprenant de la fibre d'agrume réduite en pulpe dispersée et ayant une contrainte d'écoulement dans l'intervalle de 80 à 250 Pa, de préférence de 80 à 180 Pa ;
   b) à combiner et à disperser encore le pré-mélange structurant formé dans l'étape (a) et ayant une contrainte d'écoulement de 80 à 250 Pa, de préférence de 80 à 180 Pa avec des constituants liquides de détergent en l'absence de tout autre structurant externe pour former la composition de détergent liquide aqueuse structurée en externe avec une contrainte d'écoulement dans l'intervalle de 0,3 à 1,2 Pa lorsqu'elle est mesurée à 25°C.

2. Procédé selon la revendication 1, dans lequel le pré-mélange structurant formé dans l'étape (a) présente une contrainte d'écoulement dans l'intervalle de 80 Pa à 160 Pa, de préférence de 80 Pa à 150 Pa, et bien mieux encore de 100 Pa à 150 Pa, lorsqu'elle est mesurée à 25°C

3. Procédé selon l'une quelconque des revendications précédentes, dans lequel le pré-mélange structurant formé dans l'étape (a) comprend de 1,5 à 3 % en masse de fibres d'agrumes, de préférence environ 2 % en masse.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition de détergent formée

est une composition de détergent de lessive.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel la composition comprend au moins 0,1 % en masse de particules de non-argile, de préférence au moins 0,2 % en masse de particules de non-argile.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel les particules de non-argile sont à choisir dans le groupe comprenant : des indices visuels, des capsules de parfum, des additifs de soin et des ingrédients nettoyants.

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel les particules de non-argile sont des capsules de parfum.

8. Composition de détergent liquide isotrope aqueuse structurée en externe ayant une contrainte d'écoulement dans l'intervalle de 0,3 Pa à 1,2 Pa à 25°C, comprenant :

i) au moins 10 % en masse d'eau ;
ii) au moins 3 % en masse de tensioactif détersif ;
iii) au moins 0,01 % en masse de particules de non-argile ; et
iv) un structurant externe,

caractérisée en ce que le structurant externe consiste essentiellement en de 0,05 à 0,125 % en masse de fibre d'agrume et la composition peut être obtenue par le procédé selon l'une quelconque des revendications précédentes.

9. Composition de détergent pouvant être obtenue selon la revendication 8, dans laquelle la contrainte d'écoulement de la composition de détergent se trouve dans l'intervalle de 0,30 Pa à 1,0 Pa, de préférence dans l'intervalle de 0,35 Pa à 0,85 Pa lorsqu'elle est mesurée à 25°C.

10. Composition de détergent pouvant être obtenue selon la revendication 8 ou 9, dans laquelle la fibre d'agrume est présente dans la composition de détergent dans un intervalle de 0,06 % en masse à 0,10 % en masse, de préférence de 0,06 % en masse à 0,09 % en masse, et encore mieux de 0,07 % en masse à 0,08 % en masse.

REFERENCES CITED IN THE DESCRIPTION

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