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(54) **Aqueous hand dish-wash compositions comprising surfactant and amylase**

(57) A hand dish-washing liquid composition comprising greater than 9 wt% surfactant and less than 1.5 wt% alpha-amylase enzyme based on specific protein content of the enzyme, wherein the weight ratio of surfactant to alpha-amylase is less than 30:1.

EP 2 883 949 A1

Description**Technical Field**

5 [0001] This invention relates to aqueous liquid hand dish-wash compositions comprising surfactant and amylase enzyme.

Background

10 [0002] Alpha-Amylase is an enzyme EC 3.2.1.1 that hydrolyses alpha bonds of large, alpha-linked polysaccharides, such as starch and glycogen, yielding glucose and maltose. It is the major form of amylase found in humans and other mammals. It is also present in seeds containing starch as a food reserve, and is secreted by many fungi.

[0003] An alpha-amylase called "Termamyl", sourced from *Bacillus licheniformis*, is used in some detergents, especially dishwashing and starch-removing detergents. "Termamyl" is a heat-stable alpha-amylase produced by a genetically-modified strain of *Bacillus licheniformis*. The stability of the enzyme is, however, not related to any changes made by genetic modification which is done only to enhance production of the enzyme. The enzyme is an endoamylase which hydrolyses 1,4-alpha glycosidic linkages in amylose and amylopectin; two components of starch. Starch is therefore broken down rapidly to soluble dextrans and oligosaccharides. "Termamyl" exhibits optimum activity at about pH 7 and 90 °C.

20 [0004] "Stainzyme" is a liquid enzyme preparation containing a thermostable alpha-amylase suitable for use in detergent preparations. "Stainzyme" is produced by submerged fermentation of a genetically-modified microorganism. As with "Termamyl", the enzyme protein is not itself modified. After fermentation, the enzyme is separated from the production organism and purified. "Stainzyme" is used in detergent formulations to remove starch-based stains, e.g., from pasta, gravy and baby food. Gelatinised starch tends to stick on surfaces. "Stainzyme" degrades gelatinized starch to dextrans and oligosaccharides. It is active in the range 30 to 100 °C and between pH 7 to 11. "Stainzyme" is not very sensitive to pH, but the maximum activity is reached at pH 9. It is suitable for low-temperature, hand dish-wash processes.

25 [0005] GB1296839A (Novo Terapeutisk Laboratorium A/S) discloses an enzyme material comprising alpha-amylase produced by culture of a microorganism of the species *Bacillus licheniformis* in a nutrient medium. The enzyme may be included in cleaning compositions.

30 [0006] Amylase has been used in hand dish-wash liquids to assist with starch removal from dishes and cookware. One such hand dish-wash liquid was sold under the name "Ultra Dawn Advanced Power Liquid Hand Dishwashing Detergent" in 2011 by P&G. The declared weight ratio of surfactant to amylase was greater than 20:1 (more than 23% surfactant and less than 1% amylase). Amylase has a different CAS registry number from alpha-amylase. The number for amylase, not that for alpha-amylase, was disclosed in information about this product.

35 [0007] WO 97/36977 (P&G) suggests that it is well known that surfactant can deactivate amylase. It proposes to solve that problem by use of a different type of surfactant, namely alkyl polyglucoside in combination with more stable amylases. Example 14 on page 67 gives some liquid dishwashing detergent compositions. Example 15 on page 68 gives some hard-surface cleaning compositions. In both cases the ratios of surfactant to the special stable amylases are very high. The actual use of compositions for hand dish-washing is not disclosed.

40 [0008] WO 2002/33035 (Innu Science Canada) discloses liquid hard-surface cleaning compositions containing anionic and nonionic surfactants and an enzyme mixture, such as lipase and alpha-amylase. The lipase is always used and the amylase is optional. The ratio of lipase to amylase is not disclosed so it is impossible to calculate the ratio of surfactant to alpha-amylase. The lowest possible ratio would occur if almost no lipase were used. Claim 9 has a surfactant to lipase & alpha-amylase ratio of 30 : 1.8. Thus the ratio of surfactant to alpha-amylase is at least 16.7 : 1. The technical problem of improved cleaning is solved by repeated application and prolonged contact of the composition to floor tiles.

45 [0009] WO 98/00489 (P&G) relates to detergent compositions comprising a special dianionic surfactant together with an aluminosilicate builder; the special surfactant making up for perceived shortfalls in performance of aluminosilicate builder compared to phosphate builder used with conventional surfactant systems. On page 67 liquid detergent compositions S and T comprise alpha-amylase. These compositions were not used for hand dish-washing. The alpha-amylase used was Termamyl 60T. The low activity of the enzyme granule resulted in a ratio of surfactant to alpha-amylase granule of 7.5:1.

50 [0010] There remains a need for liquid hand dish wash compositions which provide effective removal of starch and other soils.

55 **Summary of the Invention**

[0011] According to the present invention there is provided a hand dish-washing liquid composition comprising greater than 9 wt% surfactant and less than 1.5 wt% alpha-amylase enzyme wherein the weight ratio of surfactant to alpha-

amylase compound is less than 30:1, preferably less than 25:1 and more preferably less than 20:1, based on the specific protein weight content of the alpha-amylase product used.

[0012] For highly efficient cleaning of other soils the composition may comprise at least 5 wt% anionic surfactant, preferably at least 5 wt% linear alkyl benzene sulfonate salt anionic surfactant, most preferably the sodium salt of the alkyl benzene sulfonate (NaLAS).

[0013] According to a second aspect of the invention there is provided a method of hand dish-washing a hard surface stained with starch the method comprising: diluting a composition according to claim 1 with water so that the level of surfactant is reduced from greater than 90 g/L to less than 0.4 g/L and applying the resulting dilute aqueous composition to the hard surface stained with starch and soaking the hard surface stained with starch in the dilute composition for more than 1 minute.

[0014] According to a third aspect of the invention there is provided use of a composition according to claim 1 to treat starch stains on hard surfaces by soaking the stained hard surfaces in an aqueous liquor comprising less than 0.4 g/L surfactant.

Detailed Description of the Invention

[0015] Dish means a hard surface as is intended to be cleaned using a hand dish-wash composition and includes dishes, glasses, pots, pans, baking dishes and flatware made from any material or combination of hard surface materials commonly used in the making of articles used for eating and/or cooking.

Surfactants

[0016] Surfactant (detergent active) is generally chosen from anionic and nonionic detergent actives. The cleaning composition may further or alternatively comprise cationic, amphoteric and zwitterionic surfactants.

[0017] Suitable synthetic (non-soap) anionic surfactants are water-soluble salts of organic sulphuric acid mono-esters and sulphonic acids which have in the molecular structure a branched or straight chain alkyl group containing from 6 to 22 carbon atoms in the alkyl part.

[0018] Examples of such anionic surfactants are water soluble salts of alkyl benzene sulfonates, such as those in which the alkyl group contains from 6 to 20 carbon atoms; (primary) long chain (e.g. 6-22 C-atoms) alcohol sulphates (hereinafter referred to as PAS), especially those obtained by sulphating the fatty alcohols produced by reducing the glycerides of tallow or coconut oil; secondary alkanesulfonates; and mixtures thereof.

[0019] Also suitable are the salts of alkylglyceryl ether sulphates, especially of the ethers of fatty alcohols derived from tallow and coconut oil; fatty acid monoglyceride sulphates; sulphates of ethoxylated aliphatic alcohols containing 1-12 ethyleneoxy groups; alkylphenol ethyleneoxy-ether sulphates with from 1 to 8 ethyleneoxy units per molecule and in which the alkyl groups contain from 4 to 14 carbon atoms; the reaction product of fatty acids esterified with isethionic acid and neutralised with alkali, and mixtures thereof.

[0020] The preferred water-soluble synthetic anionic surfactants are the alkali metal (such as sodium and potassium) and alkaline earth metal (such as calcium and magnesium) salts of alkyl-benzenesulfonates and mixtures with olefin-sulfonates and alkyl sulfates, and the fatty acid mono-glyceride sulfates.

[0021] The most preferred anionic surfactants are alkyl-aromatic sulfonates such as alkylbenzenesulfonates containing from 6 to 20 carbon atoms in the alkyl group in a straight or branched chain, particular examples of which are sodium salts of alkylbenzenesulfonates or of alkyl-toluene-, xylene- or phenolsulfonates, alkylnaphthalene-sulfonates, ammonium diaminonaphthalene-sulfonate, and sodium dinonyl-naphthalene-sulfonate.

[0022] When synthetic anionic surfactant is to be employed the amount present in the cleaning compositions of the invention will be used at a level of at least 5 wt%., preferably at least 10 wt%.

[0023] Nonionic surfactants tend to reduce the foam produced on use of the composition. Consumers frequently associate high foam with powerful cleaning so it may be desirable to avoid the use of nonionic surfactant altogether. For compositions where this is not an issue a suitable class of nonionic surfactants can be broadly described as compounds produced by the condensation of simple alkylene oxides, which are hydrophilic in nature, with an aliphatic or alkyl-aromatic hydrophobic compound having a reactive hydrogen atom. The length of the hydrophilic or polyoxyalkylene chain which is attached to any particular hydrophobic group can be readily adjusted to yield a compound having the desired balance between hydrophilic and hydrophobic elements. This enables the choice of nonionic surfactants with the right HLB. Particular examples include: the condensation products of aliphatic alcohols having from 8 to 22 carbon atoms in either straight or branched chain configuration with ethylene oxide, such as a coconut alcohol/ethylene oxide condensates having from 2 to 15 moles of ethylene oxide per mole of coconut alcohol; condensates of alkylphenols having C6-C15 alkyl groups with 5 to 25 moles of ethylene oxide per mole of alkylphenol; and condensates of the reaction product of ethylene-diamine and propylene oxide with ethylene oxide, the condensates containing from 40 to 80% of ethyleneoxy groups by weight and having a molecular weight of from 5,000 to 11,000.

[0024] Other classes of nonionic surfactants are: tertiary amine oxides of structure $R_1R_2R_3N-O$, where R_1 is an alkyl group of 8 to 20 carbon atoms and R_2 and R_3 are each alkyl or hydroxyalkyl groups of 1 to 3 carbon atoms, e.g. dimethyldodecylamine oxide; tertiary phosphine oxides of structure $R_1R_2R_3P-O$, where R_1 is an alkyl group of 8 to 20 carbon atoms and R_2 and R_3 are each alkyl or hydroxyalkyl groups of 1 to 3 carbon atoms, for instance dimethyldodecylphosphine oxide; dialkyl sulfoxides of structure $R_1R_2S=O$, where R_1 is an alkyl group of from 10 to 18 carbon atoms and R_2 is methyl or ethyl, for instance methyl-tetradecyl sulfoxide; fatty acid alkylolamides, such as the ethanol amides; alkylene oxide condensates of fatty acid alkylolamides; and alkyl mercaptans.

[0025] If nonionic surfactant is to be employed the amount present in the cleaning compositions of the invention will generally be at least 0.1 wt%, preferably at least 0.5 wt%, more preferably at least 1.0 wt%, but not more than 20 wt%, preferably at most 10 wt% and more preferably not more than 5 wt%.

[0026] It is also possible optionally to include amphoteric, cationic or zwitterionic surfactants in the compositions.

[0027] Suitable amphoteric surfactants are derivatives of aliphatic secondary and tertiary amines containing an alkyl group of 8 to 20 carbon atoms and an aliphatic group substituted by an anionic water-solubilising group, for instance sodium 3-dodecylamino-propionate, sodium 3-dodecylaminopropane-sulfonate and sodium N 2-hydroxy-dodecyl-N-methyltaurate.

[0028] Examples of suitable cationic surfactants can be found among quaternary ammonium salts having one or two alkyl or aralkyl groups of from 8 to 20 carbon atoms and two or three small aliphatic (e.g. methyl) groups, for instance cetyltrimethylammonium chloride.

[0029] A specific group of surfactants are the tertiary amines obtained by condensation of ethylene and/or propylene oxide with long chain aliphatic amines. The compounds behave like nonionic surfactants in alkaline medium and like cationic surfactants in acid medium.

[0030] Examples of suitable zwitterionic surfactants can be found among derivatives of aliphatic quaternary ammonium, sulfonium and phosphonium compounds having an aliphatic group of from 8 to 18 carbon atoms and an aliphatic group substituted by an anionic water-solubilising group, for instance betaine and betaine derivatives such as alkyl betaine, in particular C12-C16 alkyl betaine, 3-(N,N-dimethyl-N-hexadecylammonium)-propane 1-sulfonate betaine, 3-(dodecylmethyl-sulfonium)-propane 1-sulfonate betaine, 3-(cetylmethyl-phosphonium)-propane-1-sulfonate betaine and N,N-dimethyl-N-dodecyl-glycine. Other well known betaines are the alkylamidopropyl betaines e.g. those wherein the alkylamido group is derived from coconut oil fatty acids.

[0031] Further examples of suitable surfactants are compounds commonly used as surface-active agents given in the well-known textbooks: 'Surface Active Agents' Vol.1, by Schwartz & Perry, Interscience 1949; 'Surface Active Agents' Vol.2 by Schwartz, Perry & Berch, Interscience 1958; the current edition of 'McCutcheon's Emulsifiers and Detergents' published by Manufacturing Confectioners Company; 'Tenside-Taschenbuch', H. Stache, 2nd Edn., Carl Hauser Verlag, 1981.

Optional ingredients

[0032] The composition may include optional ingredients, such as abrasive particles and additional ingredients which aid formulation properties, stability and cleaning performance.

[0033] Magnesium sulphate is desirably included from 0.5 to 5 wt% in order to ensure the desired rheological properties are achieved.

[0034] A preservative system is also desirable, for example a mixture of CIT and MIT. BIT may also be used. The level of preservative will vary according to the expected storage temperature and the quality of raw materials. From 0.0001 to 0.1 wt% is typical.

[0035] Sodium EDTA chelant is advantageously included in the compositions at a level of 0.01 to 0.5 wt%. DMDMH (glydant) may also be included into the compositions at level of from 0.005 to 1 wt%.

[0036] When the composition contains one or more anionic surfactants, the composition may preferably comprise detergent builders in an amount of more preferably from 0.1 to 25 wt. %. Suitable inorganic and organic builders are well known to those skilled in the art. Citric acid is a preferred buffer/ builder and may suitably be included at a level of from 0.01 to 0.5 wt%.

[0037] The composition may also comprise ingredients such as colorants, whiteners, optical brighteners, soil suspending agents, deterative enzymes, compatible bleaching agents (particularly peroxide compounds and active chlorine releasing compounds), solvents, co-solvents, gel-control agents, freeze-thaw stabilisers, bactericides, preservatives, hydrotropes, polymers and perfumes.

[0038] Examples of optional enzymes include lipase, cellulase, protease, mannanase, and pectate lyase.

Viscosity

[0039] The liquid composition according to the invention preferably has a viscosity from 100 to 10,000 mPa.s, more

preferably from 200 to 8,000 mPa.s, even more preferably from 400 to 6,500 mPa.s, and still even more preferably from 800 to 5,000 mPa.s, as measured at a shear rate of 20 s⁻¹ and at a temperature of 25 degrees Celsius.

Packaging

[0040] The liquid compositions may be packaged in any suitable form of container. Preferably the composition is packaged in a plastic bottle with a detachable closure /pouring spout. The bottle may be rigid or deformable. A deformable bottle allows the bottle to be squeezed to aid dispensing. If clear bottles are used they may be formed from PET. Polyethylene or clarified polypropylene may be used. Preferably the container is clear enough that the liquid, with any visual cues therein, is visible from the outside. The bottle may be provided with one or more labels, or with a shrink wrap sleeve which is desirably at least partially transparent, for example 50% of the area of the sleeve is transparent. The adhesive used for any transparent label should preferably not adversely affect the transparency.

[0041] The invention will now be further described with reference to the following nonlimiting examples.

EXAMPLES

[0042] Where amounts of Stainzyme 12L are given it is as %wt specific protein. The relationship between the level of total protein and specific protein for this particular enzyme as supplied is given in Table 1:

Table 1

Enzyme	Total protein content	Purity (%)	Specific protein content
Stainzyme 12L	34 mg/ml	45	15 mg/ml

[0043] Cleaning was carried out using a custom Martindale Abrasion rig called a WIRA. A piece of ballerina cloth was affixed to the cleaning head which was placed in contact with a tile specially designed to simulate a dish surface and to give highly repeatable cleaning results. Cleaning was done by moving the cleaning head in a lissajous motion. Any soak time used before this cleaning process and was measured using a count-down timer before starting the abrasion rig. Cleaned tiles were rinsed with demineralised water and left to dry overnight before measuring.

[0044] Spectroscopic measurements of colour intensity L* were made using an XRite Spectrophotometer (XRite Colour i7) using a 25 mm aperture and UV excluded measurement. Measurements were taken of each tile before and after cleaning and compared to a white melamine standard tile.

$$\%SR = \frac{L^* (\text{sample}) - L^* (\text{max dirty})}{L^* (\text{unsoiled}) - L^* (\text{max dirty})} \times 100$$

[0045] Cleaning is not even across the surface so 8 measurements were taken:

1 to 4 were averaged to give %SR Max (maximum cleaning)

5 to 8 were averaged to give %SR Min (minimum cleaning)

[0046] Multiple repeat tiles were cleaned per formulation. In the following examples the mean average of %SR Min values for each formulation have been reported.

[0047] The entire test was carried out at a temperature of approximately 21 °C.

[0048] The stained melamine resin based tiles used were supplied by CFT. These tiles have low in batch variability. Use of Melamine tiles is said by CFT to give a more consistent cleaning result than real dishes.

Example 1

[0049] The composition of the liquid used (without amylase) is given in Table 1. Stainzyme 12 L amylase was added separately so that it was present during cleaning at a total protein level of 0.02 g/L, whenever used. The liquid was diluted with further demineralised water to give the concentration levels indicated in Table 2. In each test the soak time was 2 minutes and the lissajous abrasion cleaning time was 1 minute, no weight was used.

Table 1

Ingredient	wt % (100% solids basis)
Demin Water	81.800
LAS Acid	11.000
LES 1EO Na	3.500
NaOH	1.500
MgSO ₄ x 7H ₂ O	2.000
Colour	0.200
TOTAL	100.000

[0050] Data given in Table 2 shows that, for bowl wash cleaning of DM-77 mixed starch (coloured) on melamine tiles, reducing the surfactant level significantly improves cleaning performance when amylase is included in the composition. It can be seen that under these conditions a surfactant level of around 0.5 g/L is sufficient to deactivate the alpha-amylase enzyme completely.

Table 2

Ave %SR Min	1 g/L	2 g/L	3 g/L
Composition	21.61	21.45	27.98
Composition + amylase	34.72	28.09	27.13

Claims

1. A hand dish-washing liquid composition comprising greater than 9 wt% surfactant and less than 1.5 wt% alpha-amylase enzyme based on specific protein content of the enzyme, wherein the weight ratio of surfactant to alpha-amylase is less than 30:1.
2. A composition according to claim 1 comprising at least 5 wt% anionic surfactant.
3. A composition according to any preceding claim comprising at least 5 wt% linear alkyl benzene sulfonate anionic surfactant, preferably the sodium salt (NaLAS).
4. A method of hand dish-washing a dish stained with starch comprising diluting a composition according to claim 1 with water so that the level of surfactant is reduced from greater than 90 g/L to less than 0.4 g/L and applying the resulting dilute aqueous composition to a surface of the dish stained with starch and soaking the dish stained with starch in the dilute composition for more than 1 minute.
5. Use of a composition according to claim 1 to treat starch stains on dish surfaces by soaking the stained dishes in an aqueous liquor comprising less than 0.4 g/L surfactant.



EUROPEAN SEARCH REPORT

Application Number
EP 13 19 7060

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