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### (54) Multiphase detergent tablet

(57) Multiphase tablets comprising a first solid phase and a second solid phase are easily damaged and/or the second phase may easily break away from the remainder of the tablet. This problem is solved by including the mul-

tiphasic tablet with the second phase in one wrap which is a water soluble film. The water soluble film is preferably transparent.

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

**[0001]** The invention relates to multiphase detergent tablets, particularly for use in a domestic dishwashing machine.

**Background to the invention**

**[0002]** Detergent compositions are known. In recent years many of the laundry and machine dishwashing compositions are in the form of tablets. These are easy to handle and provide reliable dosing levels.

**[0003]** Of the commercially available tablets many comprise a plurality of layers. These layers generally have a different colour and each layer may comprise different ingredients, which are active in the sequential steps of washing.

**[0004]** For machine dishwashing, tablets are known which comprise two layers and a third composition in the form of an insert which is held within a cavity that is punched in the upper surface of the tablet. The insert has a distinguishing colour and is therewith easily recognised by consumers.

**[0005]** US 2003/119707 discloses that there are several disadvantages associated with this type of tablet. The protruding ball may easily be damaged and there is also the necessity to place the ball accurately in the cavity during the production. We have found that in use, the protruding ball easily breaks away from the cavity. This may be caused by friction during transport. It may also happen when the wrap that is surrounding the commercial tablets is opened by a consumer without paying attention to carefully avoiding scraping and/or vigorously touching the ball when the package is opened.

**[0006]** US2003/119707 addresses this problem and provides a multiphase detergent tablet comprising a first phase having a planar or generally planar upper surface, and a second phase adhered to and partially covering the upper surface.

This product and method for its preparation require a new production process and therefore are not preferred.

**[0007]** Furthermore US 2003/0050206 discloses a composition for use in a dishwashing machine in the form of a tablet, comprising at least two small parts which may be placed in the tablet, thus being surrounded by a first detergent composition that forms the main part of the tablet. This is a complicated production process. A further disadvantage of these products is the lack of a clear visible cue because the particles are on the inner side of the tablet and hence invisible.

**[0008]** Also sometimes the gluing process to put two parts together, may be imperfect, so even if a consumer is careful in opening the wrap, an insert may still fall out.

**[0009]** Therefore there is a need to provide multiphase tablets that comprise an insert, which does not easily break away.

**Summary of the invention**

**[0010]** We have found that the inclusion of a tablet comprising a first phase and a separate second phase, in a water soluble wrap, provides a tablet which is stable and which is still attractive to the consumer.

Therefore the invention relates to a detergent tablet comprising a first solid phase and a second solid phase, wherein the tablet is wrapped in a water-soluble film.

**[0011]** In a further aspect the invention relates to the use of this tablet in a machine dishwashing process.

**Detailed description**

**[0012]** In the context of the invention the terms "wrap" and "film" are used interchangeably.

**[0013]** The tablets according to the invention are suitable for use in any application such as laundry washing or machine dishwashing processes. The invention was found especially suitable for application in a machine dishwashing process because this process benefits highly from the option to include benefit agents for the rinse cycle in a second phase.

**[0014]** The tablet according to the invention comprises at least two phases. Both phases are solid phases. Optionally the tablet comprises further phases, which may be solid, smooth, gel-like or liquid.

**[0015]** The first solid phase is also referred to as the tablet body. The tablet body according to the invention is a solid, which can, for example, be prepared by compression or melting. Preferably the tablet body is a compacted particulate composition.

**[0016]** The solid tablet body is preferably a single discrete part of the tablet but may also be composed of a limited number (for example 1 to 5) of solid parts, such as separate layers in the tablet. Preferably each of these solid parts has a weight of at least 2 grams. If reference is made to the composition or weight of the solid tablet body it is understood that this concerns the total weight and composition of these solid parts.

**[0017]** The tablet body preferably comprises ingredients such as detergency builders, surfactant and optionally bleach (activators), enzymes, chelating agent, anti-tarnishing agents for dishwashing, optical brighteners for laundry, antifoam granules, tablet disintegration aids such as swellable particles or highly water-soluble salts, perfumes, colorants or colored speckles. Some of these ingredients are described in more detail below.

**[0018]** The first phase may have any shape as long as it is possible to adhere a second phase thereto, preferably such that the second phase protrudes from a surface of the first phase. In a preferred embodiment the first phase is in a rectangular, triangular, ellipsoidal, drop-like, round or square form with a cavity in part of the surface.

**[0019]** The second phase preferably is an insert, which is at least partly placed in a mould in the surface of the first phase. Examples of such tablets are well known, e.g. the commercial tablets that contain a small ball that is

placed in a mould in the surface of a first solid tablet phase. Such tablets are for example described in US 6,730,646. This document discloses tablets having a single, substantially spherical cavity, in which is received a single, substantially spherical particle, whose external diameter is smaller than the internal diameter of the cavity. These tablets having a second, substantially spherical phase are especially suitable for application in the current invention.

**[0020]** The second phase is preferably in the form a particle. Preferably this particle is partly surrounded by a cavity in the surface of the first phase.

In a preferred embodiment the second phase is shaped like a ball and protrudes from a cavity in the surface of the tablet body.

**[0021]** Preferably the weight ratio between the tablet body and the second phase is from 40 to 1 to 1:1, more preferred from 25 to 1 to about 5 to 1.

**[0022]** The total weight of the tablet comprising the first and second phase and optionally a further phase is preferably from 10 to 30 grams.

**[0023]** In a preferred embodiment the second phase protrudes from the first phase. Preferably the protrusion of the second phase from the first phase has a diameter of from 2 to 50 mm.

**[0024]** The second phase is a solid phase. The second phase preferably comprises components that exert their function very early in the washing process before the tablet body disintegrates and dissolves, or alternatively late in the washing process, such as mainly in the rinse cycle of a machine dishwashing process.

**[0025]** A machine dishwashing process generally comprises the steps of prerinsing, main cleaning, intermediate rinsing, clear rinsing. It is preferred that the second phase comprises components that mainly evolve their function in or after the intermediate rinsing step.

**[0026]** The entire tablet is wrapped in a water-soluble wrap. It is highly preferred that the wrap is at least partly transparent such that the contents can be seen. This way the consumer does not miss out on the visible cue provided by the preferred protruding part of the tablet. Preferably the entire film is transparent. Optionally there are prints on the film, e.g. pictures or phrases.

**[0027]** The wrap preferably is a flexible wrap, contrary to solid, self standing containers. The flexible wrap preferably surrounds the tablet such that there is little room for a loosely fitted second phase, to move.

**[0028]** In a preferred embodiment, the volume of the wrap is at least 110%, more preferred from 150 to 300%, even more preferred from 150 to 250% of the volume of the tablet. The wrap should not be too loose because then it may loose functionality in keeping the second phase in place adhered to the first phase.

**[0029]** In a most preferred embodiment, the wrap is like a bag with a volume of about twice the volume of the tablet.

More preferably the wrap is tightly fit around the tablet.

**[0030]** It will be appreciated that the volume of the wrap

should be such that it does not interfere with placing it in the dispenser of a washing appliance.

**[0031]** The volume of the wrap is easily determined by filling the empty wrap with a liquid and measuring the volume of the liquid that can be put in the wrap.

**[0032]** Preferably the film material is a substantially uniform material. Such film materials can for example be produced by blowing or casting. For the purpose of the invention uniform means that the film has substantially the same composition when comparing one piece of the film with another piece of the film a distance away from the first piece. The film itself however, may consist of more than one layer.

**[0033]** The film is made of a water-soluble film material. Water-soluble material has the clear advantage that the multiphase tablet can be directly applied in the washing. The desired degree of solubilisation and strength can be achieved by matching the type of material and its thickness such that the desired solubilisation time is achieved while still maintaining the desired strength. Also preferably the film material is deformable under heating conditions.

**[0034]** Water soluble materials which may be used to form the water soluble films are widely disclosed in the literature and include, for example, polyester amides, polyvinyl alcohol, copolymers of vinyl alcohol and methacrylate, polyethylene oxide, alginates, cellulose ethers such as carboxymethyl cellulose and methylcellulose, starches and starch derivatives, gelatin and any combination of these. Especially preferred is the use of polyvinyl alcohol.

**[0035]** The water-soluble material is preferably mainly composed of poly vinyl alcohol (PVA) or of a co-polymer of poly vinyl alcohol and poly methyl acrylate (PVA-PMA).

The term poly vinyl alcohol as used herein also includes partially hydrolysed poly vinyl acetates. The water-soluble film can optionally also contain minor quantities of plasticizers, anti-foams, antioxidants, surfactants, perfumes and the like.

**[0036]** Preferably the film thickness is from 10 to 120 micrometer, more preferred from 15 to 100 micrometer, most preferred from 20 to 90 micrometer.

**[0037]** In a preferred embodiment, the second phase is fixed to the first phase with an adhesive. Alternatively, or in addition thereto, mechanical fixing is used. Optionally for fixing, compounds are selected which melt or dissolve during the wash process.

**[0038]** The composition of the second phase is preferably such that it evolves its function essentially in the clear rinsing cycle of a machine dishwashing process. Optionally the second phase is entirely surrounded by an envelope or coating which releases its contents only in or after the intermediate rinse cycle. In a preferred embodiment, such an envelope or coating incorporates at least one compound which is not or only slightly soluble at increased pH but whose solubility increases with decreasing OH- concentration and therefore with decreasing pH of the wash medium. In a typical machine dish-

washing process, the pH value changes during the process from a high pH of from 10 to 11 to a lower pH in the clear rinse cycle of from 8 to 9.

**[0039]** Suitable envelope or coating materials are for example disclosed in US-A-6,730,646.

**[0040]** The second phase preferably comprises at least one compound selected from the group comprising surfactants, antibacterial compositions, silver protection agents, fragrances, bleaches, disinfectants, odour masking agents, anti-corrosion agents, anti-film forming polymers, and enzymes. These ingredients are optionally also present in the first solid phase.

**[0041]** Materials which may be used in tablets of this invention will now be discussed in more detail.

### Surfactant

**[0042]** Anionic surfactant may typically be present in fabric washing tablets in an amount from 0.5 to 50wt%, preferably from 4 up to 30 or 40wt% by total weight of anionic surfactant based on the total weight of the tablet composition. It may be accompanied by nonionic surfactant in an amount from 3 to 20wt% by total weight of nonionic surfactant based on the total weight of the tablet composition.

**[0043]** In an automatic dishwashing composition, surfactant is overall likely to constitute from 0.5 to 8 wt%, more likely from 0.5 to 6 wt% based on the total weight of the tablet composition, and is likely to consist of non-ionic surfactant, either alone or in admixture with anionic surfactant.

**[0044]** In a preferred embodiment, the first phase comprises a surfactant and the level of surfactant is at most 10 wt% on total weight of the first phase.

**[0045]** Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Preferred examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C<sub>8</sub>-C<sub>15</sub>; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; fatty acid ester sulphonates, and primary alkyl sulphates having an alkyl chain length of C<sub>8-18</sub>.

**[0046]** It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

**[0047]** Suitable nonionic surfactant compounds which may be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide.

**[0048]** Specific nonionic surfactant compounds are alkyl (C<sub>8-22</sub>) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C<sub>8-20</sub> primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide

with the reaction products of propylene oxide and ethylene-diamine.

Especially preferred are the primary and secondary alcohol ethoxylates, especially the C<sub>9-11</sub> and C<sub>12-15</sub> primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

**[0049]** Preferred nonionic surfactants for use in machine dishwashing tablets are low to non-foaming nonionic surfactants containing ethylene oxide and/or propylene oxide residues. Examples of suitable low to non-foaming ethoxylated straight-chain alcohols which are preferred nonionic surfactants in machine dishwashing are the Plurafac LF series ex BASF, the Synperonic series ex ICI; the Lutensol® LF series, ex BASF, and the Triton® DF series, ex Rohm & Haas. Also of interest are the end-capped ethoxylated alcohols available as the SLF 18B series from BASF.

### Detergency Builders

**[0050]** Tablets according to the invention or regions thereof may contain a so-called builder which serves to remove or sequester calcium and/or magnesium ions in the water.

**[0051]** When a water-softening detergency builder is present, the amount of it generally ranges from 5 to 80 wt%, more likely from 15 to 80 wt% by total weight of detergency builder based on the total weight of the tablet composition.

**[0052]** Non-phosphorus water-soluble water-softening builders may be organic or inorganic. Inorganics that may be present include zeolites, layered silicates, alkali metal (generally sodium) carbonate; while organics include polycarboxylate polymers, such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphonates, monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono- di- and trisuccinates, carboxymethyloxysuccinates, amino polycarboxylic compounds (such MGDA (methylglycine diacetic acid carboxymethyloxymalonates)), dipicolinates, nitrilotriacetates and hydroxyethyliminodiacetates.

**[0053]** Tablet compositions preferably include polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers which have some function as water-softening agents and also inhibit unwanted deposition onto fabric from a laundry wash liquor or onto glass from an automatic dishwash liquor.

**[0054]** The category of water-soluble phosphorus-containing inorganic builders includes the alkali-metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates. Specific examples of inorganic phosphate detergency builders include sodium and potassium tripolyphosphates, orthophosphates and hexameta-phosphates. Sodium tripolyphosphate is a preferred phosphorus-containing builder for automatic dishwashing tablets. This exists in hydrated, anhydrous or partially hydrated form, and mixtures of these forms may be used

to regulate the speed of tablet disintegration and dissolution.

### Further Optional Ingredients

#### Bleach activators

**[0055]** Tablets according to the invention may contain bleach activators, also referred to as bleach precursors. Preferred examples include peracetic acid precursors, for example, tetraacetylethylene diamine (TAED), and perbenzoic acid precursors. The quaternary ammonium and phosphonium bleach activators disclosed in US 4751015 and US 4818426 (Lever Brothers Company) are also of interest. Another type of bleach activator which may be used, but which is not a bleach precursor, is a transition metal catalyst as disclosed in EP-A-458397, EP-A-458398 and EP-A-549272.

#### Bleaches

**[0056]** Preferred tablets comprise a source of hydrogen peroxide such as perborate or percarbonate, the latter being preferred.

In a preferred embodiment, the film is mainly composed of polyvinyl alcohol and the second and/or first phase comprise a bleach composition which is substantially free of sodium perborate.

#### Enzymes

**[0057]** Tablets according to the invention may contain one of the detergency enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains.

**[0058]** Suitable enzymes for fabrics include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Genencor, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark.

**[0059]** Preferred enzymes for dish washing are lipases, amylases and proteases. The enzymes most commonly used in machine dishwashing compositions are amylolytic enzymes. Preferably the composition also contains a proteolytic enzyme.

**[0060]** Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in an amount generally ranging from 0.1 to 5.0wt% by total weight of enzyme based on the total weight of the tablet composition.

#### Chelating agent

**[0061]** A chelating agent for metal ions (often referred to as a metal ion sequestrant) may be present in the

composition. If present it is preferable that the level of chelating agent is from 0.5 to 3wt% by total weight of chelating agent based on the total weight of the tablet composition. It can function to stabilise bleach against premature decomposition catalysed by transition metals and/or to inhibit scale formation.

**[0062]** Preferred chelating agents include organic phosphonates, amino carboxylates, polyfunctionally-substituted compounds, and mixtures thereof.

**[0063]** Particularly preferred chelating agents are organic phosphonates such as  $\alpha$ -hydroxy-2 phenyl ethyl diphosphonate, ethylene diphosphonate, hydroxy 1,1-hexylidene, vinylidene 1,1 diphosphonate, 1,2 dihydroxyethane 1,1 diphosphonate and hydroxy-ethylene 1,1 diphosphonate. Most preferred is hydroxy-ethylene 1,1 diphosphonate, 2 phosphono-1,2,4 butanetricarboxylic acid or salts thereof.

**[0064]** Further ingredients which can optionally be employed include anti-tarnishing agents for dishwashing,

optical brighteners for laundry, antifoam granules, tablet disintegration aids such as swellable particles or highly water-soluble salts, perfumes, colorants or coloured speckles. Fabric washing tablets may include fabric softeners and/or anti-redeposition agents such as linear sodium carboxymethylcellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose.

**[0065]** Machine dishwashing tablets may include polymers to inhibit scale formation and polymers to inhibit spotting on glassware.

**[0066]** The wrapped tablets according to the invention are preferably sold with accompanying instructions not to unwrap the tablet before use in a washing appliance.

### Method of Manufacture

**[0067]** Any suitable method may be employed. Preferably the phase of the tablet is made by convention compression methods, fixing the second phase to the first

phase, followed by inclusion in a film material by e.g. flow wrap technology. The second phase is for example made by compression or by methods such as those disclosed in US 6730646.

### Examples

#### Example 1

**[0068]** A rectangular tablet, as described by the composition below, weighing 19.6 g is prepared by stamping. The stamping mold is designed to produce a hemispherical recess of 14 mm diameter in the middle of one of the faces of the tablet.

## Tablet Composition and preparation:

Ingredient	Weight %
Sodium tripolyphosphate	49
Sodium carbonate	12.4
Sodium percarbonate	9.4
Polyethylene glycol 6000	8.0
Sodium disilicate	6.5
Protease	3.0
Nonionic surfactant	3.0
Amylase	2.5
TAED	2.7
Water&Minors	Balance to 100%

## [0069] Insert Composition and Preparation:

Ingredient	Weight %
Nonionic surfactant	45.0
Polyethylene glycol 20000	45.0
pH Sensitive polymer	4.0
Polyvinylalcohol	4.0
Water and minors	2.0

[0070] A spherical insert of approximately 12 mm diameter and weighing about 0.9 g is prepared by first forming a spherical core comprising an equal weight mixture of nonionic surfactant and polyethylene glycol of 20000 molecular weight. This sphere is coated with polyvinyl alcohol applied as an aqueous solution and then subsequently coated with an aqueous solution of a polymer whose solubility is pH sensitive. The method is analogous to that specified in US 6,730,646.

## Assembly of tablet and Flow wrap:

[0071] A drop of glue (for e.g. melted polyethylene glycol) is placed in the hemispherical cavity of the tablet, prepared as described above, and the spherical insert is placed in the cavity and left undisturbed until the insert is fixed to the tablet. The spherical insert slightly protrudes above the planar face of the tablet.

[0072] The tablet is then flow wrapped in a transparent water soluble film comprised of polyvinyl alcohol using standard flow wrap technology which is well known in the art.

Example 2

## [0073] Tablet Composition and Preparation:

Ingredient	Weight %
Sodium tripolyphosphate	49
Sodium carbonate	12.4
Sodium percarbonate	9.4
Polyethylene glycol 6000	8.0
Sodium disilicate	6.5
Protease	3.0
Nonionic surfactant	3.0
Amylase	2.5
TAED	2.7
Water&Minors	Balance

[0074] A rectangular tablet, as described by the composition above, weighing 20.7 g is prepared by stamping. The stamping mold is designed to produce a cylindrical cavity of 21 mm diameter and 8 mm depth in the middle of one of the faces of the tablet.

## [0075] Insert Composition and Preparation

Ingredient	Weight %
Sodium tripolyphosphate	50.0
Polyethylene glycol	5.0
Sodium carbonate	20.0
Polymers	10.0
Water and minors	15.0

[0076] A cylindrical inset of approximately 19 mm diameter, 7 mm tall and weighing about 2.4 g is prepared by stamping the composition described above.

## [0077] Assembly of tablet and Flow wrap:

[0077] A drop of glue (for e.g. melted polyethylene glycol) is placed in the cylindrical cavity of the tablet, prepared as described above, and the cylindrical insert is placed in the cavity and left undisturbed until the insert is fixed to the tablet. The insert is planar with or slightly below the surface of the tablet. The tablet is then flow wrapped in a water soluble film comprised of polyvinyl alcohol using standard flow wrap technology which is well known in the art.

Comparative example 3

## [0078] Tablet Composition and Preparation:

Ingredient	Weight %
Sodium tripolyphosphate	49
Sodium carbonate	12.4
Sodium percarbonate	9.4
Polyethylene glycol 6000	8.0
Sodium disilicate	6.5
Protease	3.0
Nonionic surfactant	3.0
Amylase	2.5
TAED	2.7
Water&Minors	Balance

**[0079]** A rectangular tablet, as described by the composition above, weighing 20.7 g is prepared by stamping. The stamping mold is designed to produce a cylindrical cavity of 21 mm diameter and 8 mm depth in the middle of one of the faces of the tablet.

**Claims** 25

1. Detergent tablet comprising a first solid phase and a second solid phase, **characterised in that** the tablet is wrapped in a water soluble film. 30
2. Detergent tablet according to claim 1 wherein the water soluble film is at least partly transparent.
3. Detergent tablet according to any of claims 1-2 wherein the first solid phase comprises a cavity and wherein the second solid phase is a particle, which is partly surrounded by the cavity. 35
4. Detergent tablet according to any of claims 1-3 wherein the second phase is fixed to the first phase with an adhesive. 40
5. Detergent tablet according to any of claims 1-4 wherein the first phase comprises a surfactant and the level of surfactant is at most 10 wt% on total weight of the first phase. 45
6. Detergent tablet according to claim 5 wherein the second phase comprises substances that have their main function in the rinsing cycle of a dishwashing machine. 50
7. Detergent tablet according to claim 5-6 wherein the film is mainly composed of polyvinyl alcohol and wherein the second and/or first phase comprise a bleach composition which is substantially free of sodium perborate. 55



DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (IPC)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
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CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 05 07 6980

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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