EUROPEAN PATENT APPLICATION

Detergent compositions in tablet form

The invention provides a detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, characterised in that a barrier layer formed from a hydrated polymeric material is interposed between the semi-solid region and the solid tablet body.

The barrier layer according to the invention has superior stability and appearance, as well as good barrier properties.
Description

Field of the Invention

[0001] This invention relates to detergent compositions in the form of shaped bodies, usually referred to as tablets, which are suitable for use in automatic dishwashing or laundry, which are made by a process involving compaction of a particulate composition, and which include a semi-solid region.

Background and Prior Art

[0002] Detergent compositions in tablet form have advantages over powdered products in that they do not require measuring and are thus easier to handle and dispense into the washload.

[0003] Tablets of a cleaning composition are generally made by compressing or compacting a quantity of the composition in particulate form.

[0004] Tablets comprising two or more separate regions have also been described. For example WO 00/61717 describes a detergent tablet which is characterised in that at least part of its outer surface is semi-solid. WO 99/24549 describes a detergent tablet comprising a compressed solid body and a non-compressed gelatinous portion mounted in a mould of said body.

[0005] A problem with detergent tablets which include a semi-solid region in contact with a tablet body of, for example, solid compacted particulate material is the tendency of the semi-solid to migrate into the tablet body. As well as being aesthetically unattractive, such migration may compromise tablet performance by causing chemically incompatible components to mix.

[0006] The present inventors have found that this problem can be solved by providing a barrier layer of hydrated, film-forming, water-soluble polymeric material between the semi-solid region and the tablet body.

[0007] The barrier layer according to the invention has superior stability and appearance, as well as good barrier properties.

Summary of the Invention

[0008] The invention provides a detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, characterised in that a barrier layer formed from a hydrated polymeric material is interposed between the semi-solid region and the solid tablet body.

Detailed Description of the Invention

Semi-Solid Region

[0009] The semi-solid region of the tablet is preferably a separate layer of the tablet. However the region could also have other forms, such as a core or insert.

[0010] The semi-solid region is preferably a single discrete part of the tablet but may also be a limited number (for example say 1 to 5) discrete semi-solid parts. Preferably each of these semi-solid parts are at least 1 gram, also preferably each of these semi-solid parts is substantially of the same composition. If reference is made to the composition or weight of the semi-solid region it is understood that this concerns the total weight and composition of these semi-solid parts.

[0011] The term "semi-solid" refers to compositions which are one the one hand solid enough to retain their shape at ambient temperature but which are not completely solid.

A suitable test to check if a composition can be considered as semi-solid is described and illustrated in WO03/104380. In this test, a cylindrical tablet of the composition to be tested with a diameter of 45 mm and a height of 20 mm is compressed radially between the plates of a material testing machine until the tablet fractures. At the starting position, the plates contact the tablet but do not apply force to it. Force is applied to compress the tablet, with the vertical speed of the upper plate being 25 mm/minute. The testing machine measures the applied force (F), and also the displacement (x) of the plates towards each other as the tablet is compressed. The distance (γ) between the plates before force is applied, which is the diameter of the tablet, is also known. At failure, the tablet cracks and the applied force needed to maintain the displacement drops. Measurement is discontinued when the applied force needed to maintain the displacement has dropped by 25% from its maximum value. The maximum force is the force at failure (Ff). The break energy is the area under the graph of force against displacement, up to the point of break. It is given by the equation:

$$E_b = \int_{0}^{x_f} F(x) \, dx$$

in which $E_b$ is the break energy in mjuoles, $x$ is the displacement in metres and $F$ is the applied force in Newtons at displacement $x$ and $x_f$ is the displacement at failure.

[0012] Semi-solid compositions are characterised by a ratio of $F_f$ to $E_b$ of less than 1.0, more preferably from 0.1 to 0.9, most preferably from 0.2 to 0.6, while traditional tablets of compacted particulate materials are generally characterised by a ratio of $F_f$ to $E_b$ of more than 1, more generally more than 1.25, or even more than 1.5, up to for example 6.

[0013] Preferably the semi-solid region comprises from 20 to 80 wt% of surfactants (based on the total weight of the semi-solid region), more preferably from 30 to 70 wt%, most preferably from 40 to 55 wt%.

[0014] Preferred surfactants for the semi-solid region
include nonionic surfactants, for example at a level of 30 to 50 wt% based on the weight of the semi-solid region.  

**[0015]** The semi-solid region may preferably also comprise soap, for example at a level of 0.1 to 10 wt% based on the weight of the semi-solid region.  

**[0016]** Suitably the semi-solid region of the tablet comprises diluent materials such as polyethylene glycol or (mono-) or (di-) propylene glycol. Di-propylene glycol is a preferred diluent material. Preferably the level of these diluents is from 10 to 70 wt %, more preferably from 15 to 60 wt%, most preferably from 40 to 55 wt% based on the weight of the semi-solid region.  

**[0017]** The semi-solid region preferably comprises no or only low levels of water. Preferably the level of water is less than 20 wt% based on the weight of the semi-solid region, more preferably less than 15 wt%, most preferably from 2 to 10 wt%.  

**[0018]** Preferably the total weight of surfactants in the semi-solid region is from 0.01 to 20 grams, more preferably from 0.1 to 10 grams, most preferably from 0.5 to 2 grams.  

**Solid Tablet Body**  

**[0019]** 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.  

**[0020]** 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 10 grams, also preferably each of these solid parts is substantially of the same composition. 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.  

**[0021]** Preferably the solid tablet body comprises no or only low levels of surfactants. Preferably the level of surfactants in the tablet body is less than 10 wt% (based on the total weight of the tablet body), more preferably from 0.1 to 5 wt%, most preferably from 0.5 to 2 wt%.  

**[0022]** The solid tablet body preferably comprises ingredients of the tablet other than surfactants. Examples of these ingredients are builders, bleach systems, enzymes etc. Preferably the builders in the tablet are predominantly present in the solid tablet body. Preferably the bleach system is predominantly present in the solid tablet body. Preferably the enzymes are predominantly present in the solid tablet body. For the purpose of this invention the term "predominantly present" refers to a situation in which at least 90 wt% of an ingredient is present in the solid tablet body, more preferably more than 95 wt%, most preferably more than 98 wt%.  

**Barrier Layer**  

**[0023]** A barrier layer formed from a hydrated polymeric material is interposed between the semi-solid region and the solid tablet body.  

**[0024]** Suitable polymeric materials for forming the barrier layer should meet a number of functional requirements, such as good water-solubility, coherent film formation and good adhesion to the semi-solid region and the solid tablet body.  

**[0025]** A solubility of at least 10gm per 100gm deionised water at 20°C is desirable. Higher solubilities, such as at least 20gm per 100 gm, are preferred.  

**[0026]** A softening or melting temperature above ambient, conveniently above 35°C is desirable, better at least 40°C depending on where the tablets will be sold and used. Preferably the melting temperature does not exceed 80°C, or even 70°C, since a lower melting temperature facilitates fusion of the particles of the polymeric material during compaction.  

**[0027]** Organic polymeric materials are preferred. Examples of suitable organic polymeric materials include homopolymers and copolymers of vinyl alcohol, homopolymers and copolymers of vinylpyrrolidone, polyethylene glycols, sulphonated terpolymers of acrylic acid, methyl methacrylate, sulfophenyl methallyl ether and sodium methallyl sulfonate, and polycarboxylate polymers.  

**[0028]** Preferred organic polymeric materials are polycarboxylate polymers.  

**[0029]** Polycarboxylate polymers suitable for forming the barrier layer according to the invention are preferably polymers of unsaturated monocarboxylic acids and/or unsaturated dicarboxylic acids. Suitable monocarboxylic monomers include acrylic, methacrylic, vinylacetic, and crotonic acids; suitable dicarboxylic monomers include maleic, fumaric, itaconic, mesaconic and citraconic acids and their anhydrides. The polymers may also contain units derived from non-carboxylic monomers, preferably in minor amounts.  

**[0030]** Polycarboxylate polymers suitable for forming the barrier layer according to the invention generally have a weight average molecular weight from about 1,000 to about 400,000 Daltons.  

**[0031]** Especially preferred are homopolymers of acrylic acid and copolymers of acrylic and maleic acids. Commercially available examples of such materials are the acrylic/maleic copolymers available from BASF as the Sokalan (Trade Mark) CP series and the polyacrylates available from BASF as the Sokalan® PA Series. Specific examples are Sokalan ® CP5 and preferably Sokalan ® PA 25CI. The polymers are available as powders and granules.  

**[0032]** Mixtures of any of the above described polymeric materials may also be suitable for forming the barrier layer according to the invention.  

**[0033]** Hydration of the polymeric material is important for reducing or eliminating any tendency for the polymeric
material to absorb water from the surrounding tablet environment. This can cause swelling, cracking and discoloration leading to an unacceptable tablet appearance. This is a particular problem associated with polymeric materials having hygroscopic properties, such as the polycarboxylates described above.

[0034] Hydration of the polymeric material may be carried out by storing it in a high humidity environment prior to formation of the tablet. However it is most preferred that the hydration takes place in situ, so that formation of the tablet can be operated as a continuous process.

[0035] A particularly preferred way to achieve in situ hydration of the polymeric material is to apply compaction pressure to the tablet to admiture with a hydrated salt, followed by a compaction step of sufficient pressure to cause a release of the water of crystallisation of the hydrated salt. The water of crystallisation so released is consequently absorbed by the polymeric material to provide a barrier layer formed from the polymeric material hydrated thereby.

[0036] A barrier layer formed by the above described method will accordingly comprise hydrated polymeric material and the anhydrous salt derived from the hydrated salt minus its water of crystallisation.

[0037] The total weight of barrier layer in a tablet according to the invention will generally range from 0.1 to 15wt%, preferably from 2 to 10wt%, more preferably from 5 to 8wt% (by total weight of barrier layer based on the total weight of the tablet).

[0038] The appropriate weight ratio of polymeric material to hydrated salt for admixture to form the barrier layer according to the invention will depend on some extent on the level of water of crystallisation in the molecular formula of the hydrated salt. Generally the weight ratio of polymeric material to hydrated salt may range from 5:1 to 1:5, preferably from 2:1 to 1:2, more preferably around 1:1.

[0039] Hydrated salts suitable for forming the barrier layer according to the invention are those which release their water of crystallisation when subjected to the typical compaction pressures encountered in the manufacture of detergent compositions in tablet form. The compaction pressure used to make machine dishwashing tablets, bleach tablets or other tablets containing more than 7 to 8% by weight surfactant is likely to lie in a range from 30 to 250 Mpa (e.g. 30 to 200 kN applied to a tablet cross section of 8 to 10 mm²). The compaction pressure used to make fabric washing tablets with a surfactant content over 7% by weight is likely to be in a range from 4 to 40 Mpa (e.g. 6 to 60 kN applied to a tablet cross section of about 15cm²).

[0040] A solubility of at least 10gm per 100gm deionised water at 20°C is desirable for the hydrated salt. Higher solubilities, such as at least 20gm per 100 gm, are preferred.

[0041] Inorganic hydrated salts are preferred. Examples of suitable inorganic hydrated salts include alkali metal, alkaline earth metal or ammonium acetates, phosphates, carbonates, sesquicarbonates, phosphates, citrates, silicates, percarbonates and perborates, and mixtures thereof.

[0042] Preferred are hydrated alkali metal acetates, with sodium acetate trihydrate being particularly preferred.

Product Forms and Proportions

[0043] Tablets according to this invention are particularly suitable for laundry or for automatic dishwashing.

[0044] A tablet for laundry washing will generally contain, overall, from 5% to 50wt% of surfactant and from 5% to 80wt% by weight of detergent builder which is a water-softening agent. Water-soluble disintegration promoting particles may be present in an amount from 5% to 25% by weight of the composition.

[0045] Tablets for automatic dishwashing typically contain a high proportion of water soluble salts, such as 50% to 95% by weight, at least some of which, exemplified by sodium tripolyphosphate, sodium citrate and sodium silicate, have water-softening properties.

[0046] Materials which may be used in tablets of this invention will now be discussed in more detail.

Surfactant

[0047] 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.

[0048] In an automatic dishwashing composition, surfactant is overall likely to constitute from 0.5 to 8wt%, more likely from 0.5 to 6wt% by total weight of surfactant based on the total weight of the tablet composition, and is likely to consist of nonionic surfactant, either alone or in admixture with anionic surfactant.

[0049] 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 C10-C15; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; fatty acid ester sulphonates, and primary alkyl sulphates having an alkyl chain length of C8-18.

[0050] 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.

[0051] 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.

**[0052]** Specific nonionic surfactant compounds are alkyl (C₈₋₂₂) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C₉₋₂₀ primary or secondary alcohols and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene oxide.

**[0053]** Especially preferred are the primary and secondary alcohol ethoxylates, especially the C₉₋₁₁ and C₁₂₋₁₅ primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

**[0054]** 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 Synerpon 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 Olin.

**Detergency Builders**

**[0055]** 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.

**[0056]** When a water-softening detergent builder is present, the amount of it generally ranges from 5 to 80wt%, more likely from 15 to 80wt% by total weight of detergent builder based on the total weight of the tablet composition.

**[0057]** Non-phosphorus water-soluble water-softening builders may be organic or inorganic. Inorganics that may be present include zeolites, layered silicates, alkaline earth (generally sodium) carbonate; while organics include polycarboxylate polymers, such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphonates, monomeric polycarboxylates such as citrates, gluconates, oxysuccinates, glycerol mono- di- and trisuccinates, carboxymethylxysuccinates, carboxymethylxymalonates, dipicolinates, nitrilotriacetates and hydroxyethyliminodiacetates.

**[0058]** 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.

**[0059]** 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 hexametaphosphates. 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**

**[0060]** 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. A bleach stabiliser (heavy metal sequestant) may also be present such as ethylenediamine tetramethylene phosphate and diethylenetriamine pentamethylenephosphonate.

**Enzymes**

**[0062]** Tablets according to the invention may contain one of the detergent enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains. 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.

**[0063]** 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.

**[0064]** 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**

**[0065]** A chelating agent for metal ions (often referred to as a metal ion sequestant) 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 calcium carbonate scale formation. [0066] Preferred chelating agents include organic phosphonates, amino carboxylates, polyfunctional-ly-substituted compounds, and mixtures thereof. [0067] Particularly preferred chelating agents are organic phosphonates such as α-hydroxy-2 phenyl ethyl diphosphonate, ethylene diphosphonate, hydroxy 1,1-hexylenediamine, vinylidene 1,1 diphosphonate, 1,2 dihy-droxyethane 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. [0068] Further ingredients which can optionally be em-ployed 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. [0069] Machine dishwashing tablets may include poly-mers to inhibit scale formation and polymers to inhibit spotting on glassware.

**Method of Manufacture**

[0070] In another aspect this invention provides a method of making a detergent composition in the form of a tablet, (i.e. shaped body), in which the tablet includes a semi-solid region and a solid tablet body, characterised in that a barrier layer formed from a hydrated polymeric material is interposed between the semi-solid region and the solid tablet body. [0071] Tablets according to the invention are preferably manufactured by a process comprising the steps of:

(a) inserting a particulate composition into a tablet mould;

(b) inserting an admixture of a polymeric material and a hydrated salt into the tablet mould;

(c) co-compression of the composition to form a solid tablet body of compacted particulate composition and a barrier layer of hydrated polymeric material, and

(d) preparing a semi-solid region and applying it to the composition so that the barrier layer is interposed between the semi-solid region and the solid tablet body.

[0072] Steps (a) and (b) can be in any order, although preferably the particulate composition is first introduced into the tablet mould, followed by positioning of the admixture of polymeric material and hydrated salt in the mould.

[0073] Preferably the particulate composition is flat-tened or pre-compressed before introducing the admixture of polymeric material and hydrated salt (step (b)). Most preferably the particulate composition is pre-compressed at a force of 0.01 to 5 kN/cm² before introducing the admixture of polymeric material and hydrated salt.

[0074] Preferably the co-compression in step (c) takes place at a force of from 0.1 to 20 kN/cm². Especially if the particulate composition has been pre-compressed the co-compression in step (c) can advantageously be at a force of 0.01 to 10 kN/cm², more preferably 2 to 8 kN/cm².

[0075] The semi-solid region in step (d) may be prepared by mixing of its constituent ingredients. The pressure for preparing the semi-solid region may range from 0 to 5 kN/cm², preferably from 0.01 to 5 kN/cm².

[0076] The application of the semi-solid region in step (d) may take place by spraying or under the application of a co-compression force. Such co-compression may suitably take place at a force of from 0.1 to 20 kN/cm².

[0077] Tabletting machinery able to carry out the manufac-ture of tablets of the invention is known, for example suitable tablet presses are available from Carver and from Korsch.

[0078] The size of a tablet will suitably range from 10 to 160 grams, preferably from 15g to 60g, depending on the conditions of intended use.

[0079] The tablets may be of any shape. However, for ease of packaging they are preferably blocks of substantially uniform cross-section, such as cylinders or cuboids.

[0080] The overall density of a tablet for fabric washing preferably lies in a range from 1040 or 1050gm/litre preferably at least 1100gm/litre up to 1400gm/litre. The tablet density may well lie in a range up to no more than 1350 or even 1250gm/litre.

[0081] The overall density of a tablet for machine dish-washing or as a bleaching additive, may range up to 1700gm/litre and will often lie in a range from 1300 to 1550gm/litre.

[0082] The invention will now be illustrated by the follow-ing nonlimiting Example, in which all percentages are parts by weight based on total weight unless otherwise specified.

**EXAMPLE**

[0083] A powder composition having the ingredients as shown in Table 1 below was prepared and pre-compressed at a pressure of 0.25 kN/cm² on a Carver Laboratory press, to form 18.5 gram tablets.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium tripolyphosphate</td>
<td>69.5</td>
</tr>
<tr>
<td>Sodium Carbonate</td>
<td>3.0</td>
</tr>
<tr>
<td>Non-ionic surfactant (SLF 18B, ex Olin)</td>
<td>1.0</td>
</tr>
</tbody>
</table>

**Table 1**
In a paired comparison test, the following ingredients were added to the tablets as follows:

**Tablet 1**: 0.75 gram sodium acetate trihydrate and 0.75 gram Sokalan® PA 25Cl (ex BASF)

**Tablet 2**: 0.75 gram sodium acetate anhydrous and 0.75 gram Sokalan® PA 25Cl

The resulting products were pressed at 5 kN/cm² on the Carver Press and the products were stored at moderate conditions (i.e. 20°C and 65RH).

After two weeks storage, the layer made of the anhydrous sodium acetate (Tablet 2) had swollen, causing cracking in the tablet.

In contrast, the layer made with the sodium acetate trihydrate (Tablet 1) had not swollen. It had an attractive shiny surface and the tablet underneath did not show cracks.

A semi-solid composition having the ingredients as shown in Table 2 below was prepared by mixing, heating the mixture to 80°C and casting into moulds.

### Table 2

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Parts by weight</th>
</tr>
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<tbody>
<tr>
<td>Nonionic surfactant</td>
<td>40</td>
</tr>
<tr>
<td>Tallow soap</td>
<td>6</td>
</tr>
<tr>
<td>Di-propyleneglycol</td>
<td>48</td>
</tr>
<tr>
<td>Water</td>
<td>to 100</td>
</tr>
</tbody>
</table>

In a paired comparison test, 2 ml of the semi-solid composition was sprayed on top of each of Tablets 1 and 2 as described above.

After two weeks of storage at normal room conditions (i.e. 20°C and 65RH), the product according to the invention (i.e. using sodium acetate trihydrate so the polymer is hydrated) was stable, non-swollen and of attractive appearance. In contrast, the comparative product (i.e. using anhydrous sodium acetate so the polymer is not hydrated) had an unattractive appearance. The polymer layer was swollen, distorting the overall tablet shape, and a lot of brown discoloration spots were apparent throughout the solid part of the tablet.

### Claims

1. A detergent composition in the form of a tablet, which includes a semi-solid region and a solid tablet body, characterised in that a barrier layer formed from a hydrated polymeric material is interposed between the semi-solid region and the solid tablet body.

2. A composition according to claim 1, in which the semi-solid region comprises from 20 to 80 wt% of surfactants (based on the total weight of the semi-solid region).

3. A composition according to claim 1 or claim 2, in which the solid tablet body is a compacted particulate composition.

4. A method of composition according to any one of claims 1 to 3, in which the hydrated polymeric material is a polycarboxylate polymer.

5. A composition according to claim 4, in which the polycarboxylate polymer is selected from homopolymers of acrylic acid, copolymers of acrylic and maleic acids, and mixtures thereof.

6. A method of making a detergent composition in the form of a tablet, in which the tablet includes a semi-solid region and a solid tablet body, characterised in that a barrier layer formed from a hydrated polymeric material is interposed between the semi-solid region and the solid tablet body.

7. A method according to claim 6, in which polymeric material is applied to the tablet in admixture with a hydrated salt, followed by a compaction step of sufficient pressure to cause a release of the water of crystallisation of the hydrated salt, so that the water of crystallisation so released is consequently absorbed by the polymeric material to provide a barrier layer formed from the polymeric material hydrated thereby.

8. A method according to claim 7, in which the hydrated salt is selected from alkali metal, alkaline earth metal or ammonium acetates, sulphates, carbonates, sesquicarbonates, phosphates, citrates, silicates, percarbonates and perborates, and mixtures thereof.

9. A method according to claim 8, in which the hydrated salt is a hydrated alkali metal acetate, preferably so-
dium acetate trihydrate.

10. A method according to any one of claims 6 to 9, comprising the steps of:

(a) inserting a particulate composition into a tablet mould;
(b) inserting an admixture of a polymeric material and a hydrated salt into the tablet mould;
(c) co-compression of the composition to form a solid tablet body of compacted particulate composition and a barrier layer of hydrated polymeric material, and
(d) preparing a semi-solid region and applying it to the composition so that the barrier layer is interposed between the semi-solid region and the solid tablet body.
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<td>EP 1 239 029 A (UNILEVER PLC; UNILEVER N.V) 11 September 2002 (2002-09-11) * examples 1-3 *</td>
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<td>US 6 313 080 B1 (BOSKAMP JELLES VINCENT ET AL) 6 November 2001 (2001-11-06) * column 11; figure 3 *</td>
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<td>A</td>
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The present search report has been drawn up for all claims.

Place of search: Munich  Date of completion of the search: 21 December 2005  Examiner: Pfannenstei n, H.
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