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(54) Detergent tablets

(57) A detergent tablet of compressed particulate material. The tablet or a region thereof contains at least one surface active agent, a builder and a water insoluble but water swellable disintegrating agent. More particularly the tablet or region thereof contains at least 5% by weight of solid surface active agent of which at least a portion is a solid non-ionic surface active agent and does not contain more than 3% by weight of liquid non-ionic surface active agent.

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Description

[0001] The present invention relates to detergent tablets for use in laundering operations, particularly but not exclusively domestic laundering.

[0002] In recent years, there has been a trend to the use of laundry detergent formulations in the form of tablets. Such tablets are more convenient to use than washing powders since they (the tablets) do not need to be "measured-out" and can easily be introduced into a domestic washing machine without spillage. The tablet may be introduced into the drum or dispensing drawer of such machine.

[0003] Such tablets are produced by compression of a particulate mixture which generally comprises a builder (which may be water soluble or water insoluble), a surface active agent system and ancillary components such as a bleach. bleach activator. enzymes etc. as well known in the art. Typically the surface active agent system will comprise (based on the weight of the tablet) at least about 5% of nonionic surface active agent and at least about 8% by weight of an anionic surface active agent.

[0004] For such tablets, it is necessary that they are able to disintegrate in water in an appropriate time scale to allow the various "active components" of the tablet to become dissolved or dispersed in the wash liquor. Thus, for example, a tablet to be introduced in the dispensing drawer of a domestic washing machine must be capable of dissolving in the relatively short time during which water in introduced into that drawer. For a tablet to be introduced into the drum of a washing machine, a longer dissolution time will be acceptable.

[0005] In order to control the rate at which a tablet disintegrates, it is known to incorporate a disintegrating agent. Thus, for example, various substances which have high solubility in water may be used for this purpose. Such water soluble disintegrant arc used particularly for tablets which are to be introduced into the drum of a domestic washing machine. It is also known to use disintegrants which are insoluble in water but which swell in contact therewith. Such water insoluble materials tend to be used for tablets are to be loaded into the dispensing drawer of a washing machine.

[0006] A first aspect of the present invention relates to tablets of the latter type. i.e. incorporating a water insoluble but water swellable disintegrating agent.

[0007] According to a first aspect of the present invention there is provided a detergent tablet of compressed particulate material, said tablet or a region thereof containing at least one surface active agent, a builder and a water insoluble but water swellable disintegrating agent wherein the tablet or region thereof contains at least 5% by weight of solid surface active agent and does not contain more than 3% by weight of liquid non-ionic surface active agent.

[0008] Tablets in accordance with the first aspect of the invention may have the composition as defined in the previous paragraph throughout the tablet. Alternatively this composition may be provided in a region of the tablet, e.g. a layer.

[0009] The term liquid surface active agent as used herein means a surface active agent having a viscosity of less then 1 Pa s at 20°C. Preferably any non-ionic liquid surface active agent in the tablet (or region thereof) has a viscosity of less then 0.5 Pa s at 20°C.

[0010] We have found that improvements in the disintegration of tablets containing a water insoluble but water swellable disintegrating agent may be obtained by controlling the amounts of solid surface active agent and liquid non-ionic surface active agent (which will be present in an absorbed and/or adsorbed form on solid components of the tablet or region thereof). More particularly, the improvement is obtained by ensuring that the amount of liquid non-ionic surface active agent present in the tablet (or region thereof) does not exceed 3% by weight of the tablet or region whilst ensuring that there is at least 5% by weight of solid surface active agent. By the term "does not exceed 3% by weight" we cover the possibility that the tablets (or region thereof) does not contain any liquid non-ionic surface active agent.

[0011] In particular, the present invention renders it possible to produce tablets which are intended for dosing into the dispensing draw of a domestic washing machine and which dissolve rapidly therein during the time that water is passed in to the dispensing draw. Tablet formulations may be produced which leave a residue of less than 1% by weight of the tablet in the draw after the addition of water thereto has terminated. The first aspect of the invention is however also applicable to tablets which arc intended to be loaded into the drum of a domestic washing machine.

[0012] It is particularly preferred in accordance with the first aspect of the invention that (prior to compaction of the tablet or region thereof) the disintegrant has an average particle size of 30μm to 1500μm. For example, the disintegrant may have a particle size of 50μm to 500μm, preferably 50μm to 400μm, more preferably 100μm to 300μm, and even more preferably 300μm to 350μm. Alternatively, the disintegrant may have a particle size of 300μm to 1200μm, e.g. 500μm to 1200μm, more preferably 600μm to 1000μm, and even more preferably 800μm to 1000μm. Typically the amount of the disintegrant present in the tablet (or region thereof) will be in the range 0.5% to 12% (e.g. 0.5% to 9%) by weight. more preferably 1% to 5% by weight. If the disintegrant has a particle size of 50 to 500μm it is preferably present in an amount of 0.5% to 2.5%. If the disintegrant has a particle size of 300 to 1200μm (e.g. 500μm to 1200μm) it is preferably present in an amount of 3% to 12%.

[0013] Most preferably the disintegrant is a cellulose based material. Such cellulose based material may for example comprise both crystalline and amorphous cellulose. Examples of suitable materials arc disclosed, for example, in WO-A-9855575 (Henkel), WO-A-9840462 (Herzog). The cellulose may be a cross-linked modified cellulose e.g. AC-DI-

SOL and/or may comprise micro crystalline cellulose fibres (e.g. HANFLOC).

[0014] The cellulose based material may be a cellulose derivative which may be cross-linked. e.g. a cross-linked carboxymethyl cellulose.

[0015] A particularly suitable disintegrant for use in the invention is available under the trade marks Heweten 800G (ex J.Rettenmeyer & Sohne), Norasol 771 (ex Rohn & Haas) and NILYN (ex FMC).

[0016] The disintegrant may be a cellulose derivative, for example a sodium carboxymethyl cellulose. Examples include COURLOSE and NYMCEL.

[0017] Further examples of disintegrants which may be used include various starches such as potato, rice. corn or maize starch. The disintegrant may be a starch derivative, e.g. carboxymethyl starch such as available under the trade mark PRIMOGEL or a sodium starch glycolate such as available under the trade mark EXPLOTAB.

[0018] It is also possible for the disintegrating agent to be a clay. Such clays are generally of the "lamellar type" and may for example be a smectite such as a Laponite, Bentonite, Montmorrillonite, Hectorite or Saponite. For example, the clay may be a Sodium Montmorrillonite, a Sodium Hectorite, a Sodium Saponite, a Calcium Montmorrillonite or a Lithium Hectorite.

[0019] Furthermore, it is possible for the disintegrating agent to be a synthetic polymer, for example a cross-linked polyvinyl pyrrolidone, POLYPLASDONE XL or KOLLIDON XL.

[0020] The disintegrant may be a polyacrylate or derivative thereof.

[0021] Mixtures of the above disintegrants may be used.

[0022] It will be appreciated that certain of the abovedescribcd disintegrants will provide additional benefits during a laundering operation, e.g. a clay may contribute to fabric softening properties and synthetic polymers may act to prevent deposition of dyes.

[0023] As indicated, it is feature of the first aspect of the invention that the tablet (or region thereof) does not contain more than 3% by weight of liquid non-ionic surface active agent. Tablets which do not contain any liquid non-ionic surface active agent are within the scope of the invention but it is generally preferred that the tablet contains at least 0.5% by weight of liquid non-ionic surface active agents.

[0024] The tablet may contain more than 2.5% by weight of liquid non-ionic surface active agent. Certain tablets may contain preferably not more than 2%, even more preferably not more than 1.75% and most preferably not more than 1.5% by weight liquid non-ionic surface active agent. Other tablets may contain 1 to 2.5%, e.g. 1.5 to 2.5% by weight of liquid non-ionic surface active agent.

[0025] Accordingly to a second aspect of the present invention according to which there is provided a detergent tablet of compressed particulate material, said tablet or a region thereof containing at least one surface active agent and a builder wherein the tablet or region thereof contains at least 5% by weight of solid surface active agent of which at least a portion is provided by a solid non-ionic surface active agent and not more than 1.5% by weight of liquid non-ionic surface active agent.

[0026] Tablets in accordance with the second aspect of the invention may include disintegrant. e.g. a water soluble disintegrant as described above or a water soluble disintegrant (i.e. a highly water soluble salt). Tablets in accordance with the second aspect of the invention may have the composition as defined in the previous paragraph throughout the tablet. Alternatively this composition may be provided in a region of the tablet, e.g. a layer.

[0027] Tablets in accordance with the invention may be produced by admixing the solid surface active agent (including the solid non-ionic surface active agent), liquid non-ionic surface active agent (if any) and other components of the formulation e.g. disintegrant builder, enzymes bleach. etc and compacting the resultant formulations. Alternatively the formulation (from which the tablet is compacted) may be produced by blending a solution of the (normally) solid non-ionic surface active agent with other components of the formulation such that solid non-ionic remains in the tablet (e. g. by virtue of absorption of water) but this is less preferred.

[0028] If a liquid non-ionic surface active agent is to be included in the tablet then it may for example an alcohol ethoxylate. The alcohol residue (which may be of a primary or secondary alcohol) may for example comprise 8 to 18 carbon atoms and be ethoxylated with an average of 3 to 20 moles of ethylene oxide per mole of alcohol.

[0029] Suitable liquid non-ionic surface active agents are available from ICI under the designations SYNPERONIC A3 and SYNPERONIC A7. Mixtures of the A7 and A3 active agents may also be used. Also suitable are LUTENSOL AO3 and LUTENSOL AO7 (ex BASF).

[0030] Tablets in accordance with the first and second aspects of the invention also contain at least 5% by weight at least one solid surface active agent. Preferably the amount of the solid surface active agent is in the range 5% to 25% by weight, more usually 5% to 15% by weight.

[0031] It will be appreciated that the tablets in accordance with the first and second aspects of the invention contain a relatively low amount of liquid non-ionic surface active agent, if any at all. To compensate for the low level a proportion of the solid surface active agent in the tablet (or region thereof) is a non-ionic surface active agent. The amount of solid non-ionic surface active agent may be at least 0.25%, preferably at least 0.5% and more preferably at least 1% by weight. The amount of this surface active agent may be at least 4% or 5% by weight. Most preferably the total amount

of non-ionic surface active agent present in the tablet is at least 2.5%, preferably at least 3%. more preferably at least 4% and even more preferably at least 5% of the weight of the tablet or region thereof.

[0032] Examples of solid non-ionic surface active agents which may be used in the formulation in accordance with the first aspect of the formulation include alkyl(C_{8-22})polyglycosides. The preferred glycoside employed in the present invention is a glucoside (i.e. based on glucose), functionalised with a primary alcohol (e.g. C_{12}-C_{14}). More preferably the glucoside is in the form of a polyglucoside. with a preferred degree of polymerisation of between 1-2, most preferably about 1.4.

[0033] It is preferred that solid non-ionic surface active agent is used in the form of particles or granules containing at least 20% by weight, preferably at least 30% by weight, more preferably at least 40% by weight of solid non-ionic surface active agent.

[0034] A suitable polyglycoside is available under the name Glucopon (Henkel), preferably used as Glucopon G50 granules (50% APG Glucon, 50% sulphate).

[0035] It is also preferred that at least a proportion of the solid surface active agent in the tablet (or region thereof) is provided by an anionic surface active agent. Typically the amount of such an anionic agent will generally be at least 2%, more preferably at least 4%. even more preferably at least 5%. still more preferably at least 6% and may be at least 8% by weight of the tablet (or region) thereof. Generally the amount of the anionic surface active agent will not exceed 25%, more usually not more than 20%, by weight of the tablet (or region). The anionic surface active agent may be present in an amount of 2 to 20%, more preferably 4 to 15% (e.g. 10% to 15% or 10% to 13%), more preferably 6 to 12% on the same weight basis.

[0036] It is preferred that solid anionic surface active agent is used is used in the form of particles or granules containing at least 70%, more preferably at least 80% and even more preferably at least 90% by weight of anionic surface active agent. Alternatively the anionic surfactant may he provided as an extrudate.

[0037] The anionic surface active agent may comprise at least one alkyl sulphate. most preferably a C_{8-22} alkyl sulphate. For preference, the alkyl group of the alkyl sulphate has 12-18, e.g. 8-16, carbon atoms. The alkyl sulphate may be a single compound or may comprise a mixture of alkyl sulphates of different chain lengths. For preference the alkyl groups are primary alkyl groups and preferably straight chain. The alkyl sulphate is preferably an alkali metal alkyl sulphate, the preferred alkali metal being sodium.

[0038] Suitable alkyl sulphates for use in the invention is available under the trade mark SULPHOPON, e.g. SULPHOPON 1218GF (a C_{12-18} alkyl sulphate), and MARANIL 2g (ex Cognis)

[0039] As an alternative to an alkyl sulphate, the anionic surface active agent may be an alkyl ether sulphate, preferably one in which the alkyl group has 8-22 carbon atoms. It is particularly preferred that the alkyl ether sulphate is an alkyl (C_{8-22}) ethoxylated (n=1 to 5, preferably 2 or 3) sulphate.

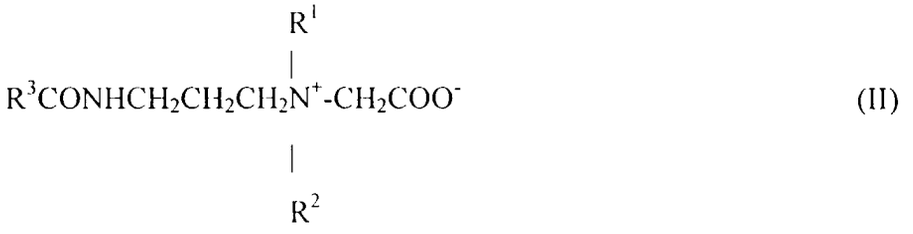
[0040] The alkyl group of the ether sulphate may be as described for the alkyl sulphate.

[0041] Further Examples of anionic surface active agents which may be used include alkylaryl sulphonates (e.g. alkylbenzene sulphonates. (e.g. Nansa HS90OF ex Albright & Wilson) alpha olefin sulphonates and ether carboxylates.

[0042] The solid surface active agent may be provided at least partially by an amphoteric surface active agent which may for example be a betaine.

[0043] Preferred betaines may be either of the formula (I) or (II).

$$R^3\text{-}N^+(R^1)(R^2)\text{-}CH_2\text{-}COO^- \qquad (I)$$

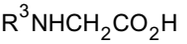


(II)

[0044] In the above formula, R^1 and R^2 may be the same or different C_{1-4} alkyl groups whereas R^3 is an alkyl group having 8-22 carbon atoms, more preferably 12 to 18 carbon atoms e.g. mixed C_{10} to C_{14}.

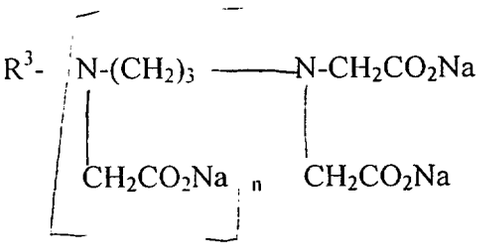
[0045] The preferred betaine for use in the tablet of the invention is cocoamidopropyl betaine (also known as coco-dimethyl acetic acid betaine (CAS Registry No.66415-29-6). Further betaines which may be used are lauryl dimethyl betaine (CAS Registry No. 683-10-3), cocoa dimethyl amidopropyl betaine (CAS Registry No.61789-40-0) and the products identified as CAS Registry Nos. 70851-07-09 and 4292-10-8.

[0046] An alternative amphoteric surface active agent for use in the tablet of the invention is a glycinate of the formula



where R^3 is as defined above.

[0047] A further glycinate which may be used is of the formula



[0048] In which R^3 is as defined above (more preferably C_{12-22}) and n is 1 to 3.

[0049] Other suitable materials are as given in chapter 1 of "Amphoteric Surfactants", e.g. Lomax Ed, Marcel Decker, New York 1996.

[0050] Tablets in accordance with the inventions may contain up to 80% by weight amphoteric surfactant, more preferably up to 5%.

[0051] It is also possible for at least a portion of the solid surface active agent to be provided by a cationic surface active agent although in this case there will generally be no anionic surface active agent present in the tablet.

[0052] Tablets in accordance with the invention also incorporate at least one builder. Generally the builder will be present in the tablets (or region thereof) in an amount of 10% to 70% by weight, more preferably 20% to 50% by weight.

[0053] It is preferred that the builder is water soluble salt and a wide range of such salts known as builders in the art may be employed in the present invention. It is particularly preferred that the builder is an alkali metal phosphate or an alkali metal phosphate or an alkali metal carbonate. A particularly preferred builder for use in the invention is sodium triphosphate for example of the type, or of a type similar to that. disclosed in EP-A-0 839 906. Alternatively the builder may be sodium carbonate.

[0054] The tablet may incorporate a bleach, for example an activated bleaching system. Such a system may comprise a hydrogen peroxide precursor (e.g. sodium percarbonate, sodium perborate monohydrate or sodium perborate tetrahydrate) together with a bleach activator.

[0055] The activator may be an N-acyl compound, particularly one having two or more N-acyl groups. Thus. for example, the activator may be tetraacetyl ethylene diamine (TAED) as conventionally used as a activator in detergent tablets.

[0056] Alternatively, the bleach activator may be an ester of a polyhydric alcohol having at least 5 carbon atoms and at least 3 hydroxyl groups esterified with C_{2-5}acyl groups, the polyhydric alcohol residue of said activator not having substituents with 6 or more carbon atoms. Such an activator may have an HLB value of at least 7. more preferably at

least 9, and even more preferably at least 11. The HLB value may be as high as 14 or 15.

[0057] The alcohol residue of the activator preferably has a maximum of 12 carbon atoms and a minimum of five hydroxyl groups esterified with C_{2-5} acyl groups. Examples of suitable alcohols are sugar and sugar derived alcohols such as sorbitol, glucitol, mannitol, glucose and sucrose.

[0058] For preference, the acyl groups in the activator are aliphatic acyl groups. It is preferred that the acyl group has two or three carbon atoms and is most preferably the acetyl group.

[0059] Specific examples of bleach activator which may be used in the tablets of the invention include hexa acetyl sorbitol. hexa acetyl mannitol, penta acetyl glucose and octa acetyl sucrose. Particularly preferred are hexa acetyl sorbitol and hexa acetyl mannitol which may be used in admixture, e.g. as disclosed in EP-A-0 525 239. Further Examples are compounds having nitrogen atoms in the basic carbohydrate skeleton. e.g. the peracetylated forms of N-methyl gluxconamide, N-methyl glucamine and glucopyronosyl amine.

[0060] Further details of the activators are disclosed in EP-A-0 869 170.

[0061] The amount of bleach activator incorporated in the tablet of the invention will generally be in the range of 0.5% to 10% by weight of the total formulation, more preferably 1% to 8% and even more preferably 2% to 4% on the same basis.

[0062] The preferred bleaching system for use in the invention comprises a hydrogen peroxide precursor compound and the bleach activator as defined above which is capable of reacting with the hydrogen peroxide to generate a peracid. The hydrogen peroxide precursor compound may, for example, be an inorganic persalt e.g. a perborate (in the monohydrate and/or tetrahydrate form), a percarbonate or a persulphate. The alkali metal salts of these compounds are preferred, particularly sodium and potassium salts. Alternatively the bleaching agent may be a urea-hydrogen peroxide complex.

[0063] The amount of hydrogen peroxide precursor compound present in the formulation of the invention is preferably such as to provide 0.5% to 3% by weight active oxygen, especially 1.0% to 2.5% by weight.

[0064] The tablet may incorporate a fabric softening agent which may for example be a clay in conjunction with a surface active agent. The fabric softening clay preferably has a particle size of at least 500 μm.

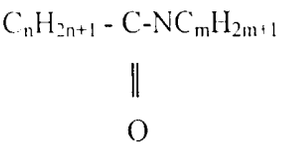
[0065] The fabric softening clay may he any such clay having fabric softening properties used in laundry detergent formulations. Such clays are generally of the "lamellar type" and are such that the layers "separate" to become deposited on the garments being washed. The clay may for example be a smectite such as a Laponite, Bentonite, Montmorrillonite, Hectorite or Saponite. For example, the clay may he a Sodium Montmorrillonite, a Sodium Hectorite, a Sodium Saponite, a Calcium Montmorrillonite or a Lithium Hectorite.

[0066] Generally the amount of clay used as a fabric softener in the detergent tablets will be 5% to 20% by weight.

[0067] The clay may be used in conjunction with a cationic and/or amide surfactant to help delamination of the clay and absorption thereof onto the garments being laundered. The cationic surfactant may for example be a quaternary ammonium salt having one long chain (e.g. C_{8-22}) alkyl group and three short chain (e.g. C_{1-4}) alkyl groups. A suitable cationic surfactant is coco trimethyl ammonium chloride. The amide surfactant may contain at least one long chain (e. g. C_{8-22}) alkyl group and may for example be stearyl stearamide. A suitable clay formulation may contain 20-30% by weight of the formulation (i.e. clay plus surfactants) of amide surfactant and 1-2% cationic surfactant.

[0068] The fabric softening agent may be an organic compound.

[0069] One class of organic fabric softening agents are amides of the formula



where n and m are the same or different and are in the range 8 to 22, more preferably 10 to 20. If the alkyl groups are branched then they preferably include a chain of at least 8 carbon atoms.

[0070] A particularly preferred amide for use in the invention is stearyl stearamide.

[0071] Alternatively, or additionally, the organic fabric softening agent may be a quaternary ammonium salt having one long chain (e.g. C_{8-22}) alkyl group and three short chain (e.g. C_{1-4}) alkyl groups. A suitable cationic surfactant is coco trimethyl ammonium chloride.

[0072] It is particularly preferred that the quaternary ammonium salt be used in combination with the above described amides in which case the quaternary ammonium salt may suitably be employed in an amount of up to 5%, more preferably 1 to 2%, by weight of the clay.

[0073] Further organic fabric softening agents which may be used include amine and/or amide functionalised silanes.

[0074] The tablet may incorporate at least one enzyme.

[0075] The enzyme may, for example, be a protease, amylase, lipase or cellulase (or mixtures thereof) such as commonly used in detergent formulations. Examples of suitable enzymes are available under the names Opticlean, Savinase, Esperase; Termamyl, Maxamayl, Lipomax, Lipolase; Celluzyme and Carezyme. The amount of enzyme incorporated in the tablet will depend on activity but will typically be 0.1 to 3%. This level is particularly suitable for Savinase 6.0T, Termamyl 60T, Celluzyme 0.7T and Lipomax.

[0076] The tablets of the invention may be mass-produced on a number of tabletting machines. Models that may be used include the Europharma Machinery (UK) generally work by having a rotating circular turret with arrays of punches that compress the tablets from above and below. Tablets may be produced that are single or dual or multi- layer or of the tablet-in-tablet type and variations thereof. The cycle for producing dual layer tablets consists of filling the die with the powder that will make up one of the layers, followed by filling of the powder of second layer.

[0077] Machines specially designed for dual layer operation usually have a small amount of pre-compression between filling the die with the powders of the first and second layers. This gives a sharper definition between the two layers which may be more aesthetically pleasing, particularly if the two layers are of different colours.

[0078] The press should have a control to regulate the applied force used in the main compression. The applied pressure should typically be about 10 to 120KN for a 44mm tablet produced at a rate of greater than 20000 tablets per hour. The pressure applied is a crucial part of the tabletting operation as inadequate pressure will gives a tablet which dissolves too slowly. The tablet strength may be monitored by use of equipment to measure its breaking strength such as the Holland CT5 automatic compression tester (see below).

[0079] It will he appreciated that the tablet may incorporate additional components as conventionally included in laundry detergent formulations. One Example of such an additional component is a soap which may be used in an amount up to 5% by weight as a processing aid. Further Examples include anti-foam agents, sequestrants (e.g. of the phosphonate type), whiteness maintenance agents (e.g. CMC, polyoxyethylene terephthalate, polyethylene terephthalate), colourants (e.g. dyestuffs), perfume, flow control agents (e.g. a sulphate) flow enhancer (e.g. a zeolite), pH regulators (e.g. a carbonate or bicarbonate), anti-corrosion agents, dye transfer inhibitors (e.g. PVP) and optical brighteners (e.g. Tinopal CBS-X and Tinopal DMS-X). These components may, for example, each be present in amounts up to 1% by weight of the formulation.

[0080] The fabric softening agent may be an organic fabric softening agent which is nitrogen containing compound having at least a degree of positive charge on the nitrogen atom.

[0081] The invention will he further described with reference to the following non-limiting Examples.

Examples

[0082] Tablets of various compositions (see below) where produced and evaluated using the following procedures.

Beaker Test

[0083] Tablets (45mm diameter, 18mm height, 40g) are placed on an open wire stand (40m diameter, 5cm high) in a beaker containing water at 30°C. The time taken for the bulk of a tablet to fall from the stand was measured. A time of 30 seconds or less indicates suitability of the tablet for the testing in the dispensing drawer of a domestic washing machine.

Washing Machine Drawer Tests

[0084] Two tablets flat are placed on their circular faces and side-by-side, in the main (first) dispensing drawer (clean and dry) of a washing machine (drawer empty). Two washing machines are used; a Hotpoint Ultima 1200 washing machine set at 40°C normal cotton wash and a Bosch 20001 set for a 40°C wool wash. A tablet was said to have passed the drawer test if less than 1% of the tablets, by weight when dry, was left in the drawer.

Snap Test

[0085] Three point snap tests were carried out using a Holland (UK) CT5 compression tester, tablets are placed flat, on their circular face, on two bars placed 21mm apart in the load cell. A third bar then compresses the middle of the tablet from above. This is performed at ambient temperature. A 40 gram tablet should break at about 2-6Kg applied force.

Drop Test

[0086] The tablet is dropped from a height of 1 meter onto a hard surface.

Example 1

[0087] This Example demonstrates the effect of varying the amount of liquid non-ionic surface agent whilst maintaining the total non-ionic surfactant constant in a tablet containing a water insoluble but water swellable disintegrating agent.

Tablets of the following compositions 1A - 1D were prepared				
Component	Invention 1A	Comparative 1B	Comparative 1C	Invention 1D
STP	41.5	41.5	41.5	41.5
Sodium Disilicate Granules	5.00	5.00	5.00	5.00
C_{12-18} Primary Alkyl Sulphate	10.00	10.00	10.00	10.00
Glucopon 50 G	2.00	1.00	0.00	3.00
T.A.E.D	3.00	3.00	3.00	3.00
CMC Disintegrating	1.50	1.50	1.50	1.50
Agent	0	0	0	0
Alkyl Ethoxylate (7EO)	3.00	4.00	5.00	2.00
Minor components	1.62	1.62	1.62	1.62
Na Percarbonate Disintegrating	18.00	18.00	18.00	18.00
Agent	4.50	4.50	4.50	4.50
Na Carbonate	9.88	9.88	9.88	9.88
Total %	100	100	100	100

[0088] The tablets were prepared by spraying the liquid non-ionic surfactant onto those components listed in the Table above the ethoxylate, admixing the products with those components of the formulation listed in the Table below the ethoxylate. and compacting 40g of the admixture at various pressures on a laboratory press using a die of diameter 45mm and a height of 38mm.

[0089] The tablets were then tested using the above described procedures.

Results

[0090]

(a) Composition (1A) (3% liquid non-ionic; 2% solid non-ionic)				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
3	2.18	19	Fail	Pass
4	2.94	20	Fail	Pass
5	3.52	23	Fail	Fail
6	4.79	26	Fail	Fail

Composition (1B) (4% liquid non-ionic; 1% solid non-ionic)				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
3	1.84	21	Fail	Pass
4	2.59	23	Fail	Pass
5	3.60	27	Fail	Fail

(continued)

Composition (1B) (4% liquid non-ionic; 1% solid non-ionic)				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
6	4.53	29	Fail	Fail

Composition (1C) (5% liquid non-ionic; 0% solid non-ionic)				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
3	2.69	23	Fail	Pass
4	4.05	26	Fail	Pass
5	5.37	28	Fail	Fail
6	6.36	31	Fail	Fail

Composition (1D) (2 % liquid non-ionic; 3% non-ionic)				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
3	2.25	19	Pass	Pass
4	3.36	20	Fail	Pass
5	4.24	22	Fail	Pass
6	5.87	24	Fail	Pass

[0091] The above results demonstrate that for a given compaction pressure increasing the level of liquid non-ionic surface active agent decreased the solubility of the tablet in the beaker test. increased the strength of the tablet and decreased the drawer solubility. The best results were obtained for the tablets of the invention at which the level of liquid non-ionic surface active agent was 2%. At the 3% liquid non-ionic level the tablet gave similar results in the drawer test to the 4% and 5% levels but solubility results in the beaker test were similar to those at the 2% level (and improved to those at the 4% and 5% levels). A comparison of the results at the 2% and 4% levels shows that the former gave a harder tablet with better solubility then the latter.

Example 2

[0092] This example demonstrates the effect of varying the amount of a water insoluble but water swellable tablet disintegrant in a tablet containing 1% of liquid non-ionic surface active agent.

[0093] Using the procedure of Example 1, tablets were prepared from the following compositions 2A-2G containing a bentonite clay granule (Laundrosil - ex Sud Chemie).

Component	2A	2B	2C	2D	2E	2F	2G
STP	40.5	40.5	4.0.5	40.5	40.5	40.5	40.5
Sodium Disilicate Granules	5.00	5.00	5.00	5.00	5.00	5.00	5.00
C_{12-18} Primary Alkyl Sulphate	11.00	11.00	11.00	11.00	11.00	13.00	13.00
Glucopon 50 G	2.00	2.00	2.00	2.00	2.00	2.00	2.00
Disintegrant	**0.00**	**1.00**	**2.00**	**3.00**	**4.00**	**3.00**	**4.50**
T.A.E.D	3.00	3.00	3.00	3.00	3.00	3.00	3.00
Minor Components	0.65	0.65	0.650	0.65	0.65	0.65	0.65
Alkyl Ethoxylate (7EO)	**1.00**	**1.00**	**1.00**	**1.00**	**1.00**	**1.00**	**1.00**
Enzyme	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Na Percarbonate	16.00	16.00	16.00	16.00	16.00	14.00	14.00
Laundrosil	19.36	18.36	17.36	16.36	15.36	16.36	14.86

(continued)

Component	2A	2B	2C	2D	2E	2F	2G
Total %	**100.00**	**100.00**	**100.00**	**100.00**	**100.00**	**100.00**	**100.00**

[0094] The tablets were tested as described above and the following results obtained.

Results

[0095]

Composition 2A				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
1	6.50	22	Fail	Fail
2	8.19	24	Fail	Fail
3	10.97	28	Fail	Fail
4	14.62	42	Fail	Fail
5	18.29	39	Fail	Fail

Composition 2B				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
2	6.46	19	Pass	Pass
3	8.90	21	Fail	Fail
4	10.83	24	Fail	Fail
5	14.37	28	Fail	Fail

Composition 2C				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
1	4.59	18	Pass	Pass
2	7.76	22	pass	Pass
3	9.73	25	Pass	Pass
4	11.68	28	Fail	Fail
5	13.42	32	Fail	Fail

Composition 2D				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
1	2.67	12	Pass	Pass
2	4.83	16	Pass	Pass
3	7.32	19	Pass	Pass
4	9.39	21	Pass	Pass
5	12.92	24	Fail	Fail

Composition 2E				
Pressure (Tons)	Snap (kg)	Beaker(s)	Bosch	Hotpoint
2	4.73	11	Pass	Pass
3	6.89	13	Pass	Pass
4	8.45	16	Pass	Pass
5	12.16	18	Fail	Fail

Composition 2F				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
2	6.30	17	Pass	Pass
3	8.27	19	Fail	Fail
4	10.45	22	Fail	Fail
5	13.48	24	Fail	Fail

Composition 2G				
Pressure (Tons)	Snap (kg)	Beaker (s)	Bosch	Hotpoint
2	7.07	14	Pass	Pass
3	8.28	16	Pass	Pass
4	12.82	19	Fail	Fail
5	13.84	24	Fail	Fail

[0096] The above results demonstrate that as the amount of disintcgrant is increased so is the drawer solubility of the tablet. For comparative composition 2A (no disintegrant) the tablet failed all drawer tests and displayed inferior solubility in the beaker test to the other formulations that did contain disintegrant. The dissolution times in the beaker become faster with every increase in disintegrant. As the amount of disintegrant increases, the strength of the tablet decreases slightly. The addition of increasing amounts of anionic surface active agent increases the strength of the tablet but still gives good drawer solubility

Example 3

[0097] This Example demonstrates the effect of varying the liquid non-ionic surface active agent content in a formulation which includes a fabric softener and a water insoluble disintegrant.

[0098] Using the procedures described in Example 1, tablets were prepared from the following compositions 3A-3D.

Component	3A	3B	3C	3D
STP	40.5	40.5	40.5	40.5
Disilicate Granules	5.00	5.00	5.00	5.00
C_{12-18} Primary Alkyl Sulphate	11.00	11.00	11.00	11.00
Glucopon 50 G	2.50	2.00	2.50	2.00
Disintegrant	4.50	4.50	6.00	6.00
T.A.E.D	3.00	3.00	3.00	3.00
Minor Components	0.65	0.65	0.65	0.65
Alkyl Ethoxylate (7EO)	0.50	1.00	0.50	1.00
Enzyme	1.49	1.49	1.49	1.49
Na Percarbonate	16.00	16.00	16.00	16.00

(continued)

Component	3A	3B	3C	3D
Laundrosil	14.86	14.86	13.36	13.36
Total %	100.00	100.00	100.00	100.00

[0099] The tablets were tested as previously and the following results obtained.

Results

[0100]

Composition 3A			
Pressure (Tons)	Beaker (s)	Bosch	Hotpoint
2	12	Pass	Pass
3	15	Pass	Pass
4	17	Pass	Fail
5	17	Pass	Fail
6	20	Fail	Fail

Composition 3B			
Pressure (Tons)	Beaker (s)	Bosch	Hotpoint
2	15	Pass	Pass
3	16	Pass	Pass
4	17	Pass	Pass
5	20	Fail	Fail

Composition 3C			
Pressure (Tons)	Beaker (s)	Bosch	Hotpoint
2	11	Pass	Pass
3	11	Pass	Pass
4.	13	Pass	Pass
5	16	*Pass	Fail

* Tablets compressed at 6 tons passed this test but tablets compressed at 7 tons failed.

Composition 3D			
Pressure (Tons)	Beaker (s)	Bosch	Hotpoint
2	10	Pass	Pass
3	12	Pass	Pass
4	14	Pass	Pass
5	18	*Pass	*Pass

* Failed for compression pressure of 6 tons.

[0101] It was concluded that formulations with 4.5% disintegrant varying the non-ionic level had little or no effect on beaker dissolution times, for the 6% disintegrant formulations the beaker dissolution times were fastest in the formulation with the lower non-ionic level. In the machine drawers the formulation with 0.5% disintegrant performed better in the Bosch whereas, the formulation with 1% non-ionic performed better in the Hotpoint machine, the opposite of this was true for the 6% disintegrant formulations.

Example 4

[0102] The Example demonstrates the effect of varying builder content in a tablet containing a water insoluble disintegrating whilst maintaining liquid non-ionic surface active agent constant.

[0103] Using the procedures described in Example 1, tablets were prepared from the following "non-bio" compositions 4A-4C and the "Colour Formulations" 4D-E.

Table 1

Non-Bio Formulations			
Component	4A	4B	4C
STP	41.5	39.5	43.5
Disilicate Granules	5.00	5.00	5.00
C_{12-18} Primary Alkyl Sulphate	11.00	11.00	11.00
Glucopon 50 G	2.00	2.00	2.00
Disintegrant	6.50	6.50	6.50
T.A.E.D	3.00	3.00	3.00
Minor Components	1.40	1.40	1.40
Alkyl Ethoxylate (7EO)	1.80	1.80	1.80
Na Percarbonate	18.00	18.00	18.00
Na Carbonate	9.80	11.80	7.80
Total %	100	100	100

Table 2

Colour Formulations		
Component	4D	4E
STP	46.5	48.5
Disilicate Granules	5.00	5.00
C_{12-18} Primary Alkyl Sulphate	11.00	11.00
Glucopon 50 G	2.00	2.00
Disintegrant	6.50	6.50
PVP	1.14	1.14
Minor Components	1.29	1.29
Alkyl Ethoxylate (7EO)	1.80	1.80
Enzyme	1.49	1.49
Na Bicarbonate	23.28	21.28
Total %	100	100

[0104] The tablets were tested as previously and the following results obtained.

Composition	Pressure (Ton)	Snap (Kg)	Beaker (Seconds)	Bosch	Hotpoint
4A	3	3.87	16	Pass	Pass

(continued)

Composition	Pressure (Ton)	Snap (Kg)	Beaker (Seconds)	Bosch	Hotpoint
4B	3	4.45	16	Pass	Pass
4C	3	4.74	15	Pass	Pass
4D	3	5.57	18	Pass	Pass
4E	3	5.38	18	Pass	Pass

Example 5

[0105] This Example illustrates the effect of varying amounts and/or components in various formulations containing low levels of liquid non-ionic surface active agent.

[0106] Using the procedures described in Example 1. tablets were prepared from the following formulation 5A.

Component	5A
STP	41.5
Layered Silicate	5.00
C_{12-18} Primary Alkyl Sulphate	10.00
T.A.E.D	3.00
CMC	1.50
Minor Components	1.62
Alkyl Ethoxylate (7EO)	5.00
Na Percarbonate	18.00
Disintegrant	1.25
Na Bicarbonate	12.88

[0107] Tablets were also prepared from modified formulations 5B-5D as detailed below.

5B Composition 5A with 1.5% Alkyl Ethoxylate (7EO)
5C Composition 5A with 2.5% Alkyl Ethoxylate (7EO)
5D Composition 5A with 7% Alkyl Ethoxylate (7EO)

[0108] Formulations 5B-5D were produced by adjusting the bicarbonate level to 100%.

[0109] The tablets were produced at compression pressures of 1000psi on a hand press.. The dissolution time of the tablets was measured in beaker of 30°C water. The snap hardness of the tablets was measured on a CT5 compression tester.

[0110] The following results were obtained:

Results

[0111]

Table Powder Variant	Hardness	Beaker (min/sec)
(5A)	3.62	30
(5B)	2.69	22
(5C)	3.5	27
(5D)	2.45	46

Example 6

[0112] This Example demonstrates the benefits of using the solid non-ionic surface active agent "as is" rather than

as a solution (40%).

[0113] Tablets were prepared from the following compositions 6A-6B.

	6A	6B
STP	40.75	40.75
Layered Silicate Primary Alkyl	5.00	5.00
Sulphate	13.00	13.00
Glucopon KE3515	3.00	
TAED	3.00	3.00
CMC	1.50	1.50
Glucopon 600		3.00
Na Percarbonate	16.50	16.50
Disintegrant	1.50	1.50
Laundrosil	10.00	10.00
Na Bicarbonate	0.20	0.20
Minor Components	to 100%	to 100%

[0114] Glucopon 600 is a 50% aqueous solution of alkyl polyglucoside with a C_{12} to C_{14} chain length. Glucopon KE3515 is granule containing 50% APG and 50% carboxylate carrier.

[0115] In preparing the tablets from composition 6A, the Glucopon 600 was sprayed onto the base material using a hand held plastic spray bottle. As Glucopon is too viscous until a temperature of ca. 50°C is reached, the spray bottle was immersed into near-boiling water before use. Similarly, in the preparation of tablets from composition 6B the Glucopon KE3515, the Glucopon was heated before addition to the powder in a Moulinex Masterchef 20 blender. After addition of the Glucopon, any remaining materials were added e.g. sodium bicarbonate. The mixtures were then left for 24 hours before being tableted on a hand press (600psi).

[0116] The tablets were tested and the following results obtained.

	Drop Test	Hardness	Beaker (min/sec)
9A - Glucopon (sprayed on)	Fail	3.81	2:19
9B - Glucopon (blended in)	Pass	8.47	34s

[0117] The results demonstrate that adding the solid non-ionic surface active agent in the form of a solution covering the whole composition gave a tablet that failed the drop test and had poor solubility.

Example 7

[0118] Tablets were prepared at various compression pressures from composition B save that the primary alkyl sulphate was replaced by a lauryl ether sulphate. The resultant tablets were tested and the results are shown below.

Compression	Hardness	Beaker Test
100psi	2.43kg	24 sec
1200 psi	3.45kg	25 sec
1400 psi	4.09kg	30sec

[0119] This information demonstrates that replacement of the alkylsulphate (in composition 6B) by another anionic surfactant still provides the benefits of the invention.

Claims

1. A detergent tablet of compressed particulate material, said tablet or a region thereof containing at least one surface active agent, a builder and a water insoluble but water swellable disintegrating agent wherein the tablet or region

thereof contains at least 5% by weight of solid surface active agent of which at least a portion is a solid non-ionic surface active agent and does not contain more than 3% by weight of liquid non-ionic surface active agent.

2. A tablet as claimed in claim 1 wherein the disintegrant has an average particle size of 30 μm to 1500 μm.

3. A tablet as claimed in claim 2 wherein the disintegrant has an average particle size of 50 μm to 500 μm and is preferably present in an amount of 0.5 to 2.5% by weight.

4. A tablet as claimed in claim 2 wherein the disintegrant has an average particle size of 500 μm to 1200μm and is preferably present in an amount of 3 to 12% by weight.

5. A tablet as claimed in any one of claims 1 to 4 wherein the disintegrant is a cellulose based material, e.g. cellulose derivative, starch, e.g. starch derivative, clay or synthetic polymer.

6. A tablet as claimed in claim 5 wherein the disintegrant a comprises crystalline cellulose and amorphous cellulose, is a cross-linked cellulose (preferably a cross-linked carboxymethyl cellulose), or microcrystalline cellulose.

7. A tablet as claimed in any one of claims 1 to 6 wherein the tablet or region thereof contains 0.5% to 2.5% by weight of liquid non-ionic surface active agent.

8. A tablet as claimed in claim 7 wherein the tablet or region thereof contains 1.5% to 2.5% by weight of liquid non-ionic surface active agent.

9. A detergent tablet of compressed particulate material, said tablet or a region thereof containing at least one surface active agent and a builder wherein the tablet or region thereof contains at least 5% by weight of solid surface active agent of which at least a portion thereof is a solid non-ionic surface active agent and not more than 1.5% by weight of liquid non-ionic surface active agent.

10. A tablet as claimed in claim 9 wherein the tablet or region thereof contains more than 0.5% to 1.5% by weight of liquid non-ionic surface active agent.

11. A tablet as claimed in any one of claims 1 to 9 wherein the liquid non-ionic surface active agent if present in the tablet or region thereof is an alcohol ethoxylate.

12. A tablet as claimed in any one of claims 1 to 11 wherein the tablet or region thereof contains 5% to 25% by weight of at least one solid surface active agent.

13. A tablet as claimed in claim 12 wherein the tablet or region thereof comprises at least 1% by weight of solid non-ionic surface active agent.

14. A tablet as claimed in any one of claims 1 to 13 wherein the solid non-ionic surface active agent is an alkyl (C_{8-22}) polyglycoside.

15. A tablet as claimed in claim 14 wherein the glycoside is a polyglucoside.

16. A tablet as claimed in claim 15 where the polyglucoside has a degree of polymerisation of 1 to 2.

17. A tablet as claimed in any one of claims 1 to 16 wherein at least a portion of the solid surface active agent is an anionic surface active agent.

18. A tablet as claimed in claim 17 wherein the tablet or region thereof comprises at least 5% by weight of the solid anionic surface active agent and preferably at most 25% by weight.

19. A method of producing a tablet as claimed in any one of claims 1 to 8 comprising admixing the solid surface active agent, liquid non-ionic surface active agent (if any), builder and disintegrating agent and compacting the resultant formulation to produce the tablet.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 00 30 6594

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
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P,X	DE 198 07 321 A (HENKEL KGAA) 26 August 1999 (1999-08-26) * claims 1-14; example C; table 1 * * page 7, line 9 - line 34 * ---	1,2,5-7, 9,11-14, 17,18	
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TECHNICAL FIELDS SEARCHED (Int.Cl.7)

C11D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	21 November 2000	Loiselet-Taisne, S

CATEGORY OF CITED DOCUMENTS

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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 00 30 6594

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