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(54) Title: CONTROLLED RELEASE DETERGENT TABLETS

(57) Abstract: The description relates to a controlled release detergent tablet comprising from 50 to 99% by weight of at least one active component with a detergent action and from 1 to 50% by weight of at least one hydrophobic substance. The hydrophobic substance has a melting point of between 30 and 80 °C, preferably between 55 and 75 °C. The tablet may be in the form of a coated core, where the core essentially consists of the active component and the coating essentially consists of the hydrophobic substance; alternately, the tablet may be in the form of a compressed granulate consisting of a mixture of active component and hydrophobic substance.



1

### Controlled release detergent tablets

The present invention relates to a detergent tablet with controlled release of the active component, comprising at least one active component with a detergent action and at least one hydrophobic substance, such as a fat or a wax for example.

The tablets currently manufactured in the detergent sector are very fast-dissolving and base the separation of immiscible components on the compression in layers (double or triple) or on the combination of several tablets.

The rapid solubility of the tablet may constitute a serious problem since it does not allow a homogeneous distribution of the active component within the washing environment, with the consequent risk of damaging the fabrics owing to an excessive local concentration of the active component; moreover, the need to use a dual layer system in the case of active components which are immiscible with each other constitutes a disadvantage during production.

The object of the present invention is to provide a detergent tablet which allows not only a controlled release of the active component but also, where necessary, the mixing, in a single phase, of active components which are normally immiscible with each other.

This result has been achieved now by combining, in a single tablet, the active component with a detergent action and one or more hydrophobic substances.

The invention may be implemented more effectively using hydrophobic substances having a melting point of between 30 and 80° C, preferably between 55 and 75° C.

According to a preferred aspect, the tablet comprises 50 to 99% by weight of at least one active component with a detergent action and 1 to 50% by weight of at least one hydrophobic substance (the percentages are to be regarded as being relative to the weight of the tablet).

Preferably, the active component with a detergent action is present in an amount equal to 60-99% by weight, even more preferably in an amount equal to 65-95% by weight; the hydrophobic substance is present in an amount equal to 1-30% by weight, even more preferably in amount equal to 1-15% by weight.

The method of preparing the tablets according to the invention is based on the following principles:

- preparation of granulate by means of mixing of the active component(s) in the form of powder with fats, waxes or paraffins in the melted state, cooling and compression;
- coating in a pan, by means of a sprayer, of granulates or ground powders with fats, waxes or paraffins in the melted state and then compressing thereof, either as such or mixed with filler powders or other active components;
- simple mixing of active powders with fats, waxes or paraffins in the pulverized state and compressing of the mixture in the form of normal tablets or multilayered tablets.

With the technology according to the present invention it is possible to obtain:

- slow release tablets;
- tablets having, mixed therein, active components
   which may interact if placed in close contact, such

as, for example, sodium percarbonate, sodium perborate and TAED (tetra acetyl ethylene diamine);

• tablets which are required to release immediately into the solution certain active components and release slowly other active components (multilayered tablets).

The active components which may be used with the present technology are:

- the oxidizers percarbonates, persulphates, perborates;
- TAED or other active components which interact with the released oxygen;
- detergents and softener, phosphates, polyphosphates, EDTA, citric acid, sodium bicarbonate, sodium carbonate, alkyl sulphonates, trichloroisocyanuric acid, potassium dichloroisocyanurate, chloramine-T, phosphates, potassium pyrophosphates, tripolyphosphates, silicate, sodium silicate, xylene sulphonate, toluene sulphonate, cetrimide, sodium sulphonate, alkylaryl sulphonate, monoisopropanolamide, sodium metasilicate, sodium lauryl sulphate, non-ionic surfactants, anionic surfactants, cationic surfactants, protease, lipase,  $\alpha$ -amylase, amylase, borax and sodium sulphate.

The hydrophobic retarding agents may be:

- hydrogenated vegetable fats (for example, palm, castor bean, etc.) with a melting point of between 30°C and 80°C;
- waxes with a melting point of between 30°C and 80°C;
- animal and vegetable fats with a melting point of

between 30°C and 80°C;

• paraffins with a melting point of between  $30^{\circ}\text{C}$  and  $80^{\circ}\text{C}$ .

The tablet may contain filler excipients such as, for example, carbonates, phosphates, sulphates and the like, cellulose and cellulose derivatives, sugars, amides and proteins. These excipients are normally contained in amounts of between 0.1 and 20% by weight of the tablet, preferably between 0.5 and 15% by weight, even more preferably between 1.1 and 10% by weight.

For the purposes of the present invention, moreover, the term "essentially consisting of" is understood as meaning that the tablet, in addition to the active component and the hydrophobic substance, may contain minimum amounts (and in any case not more than 20% of the weight of the tablet) of the abovementioned filler excipients, such that active components, hydrophobic substances and any excipients form 100% of the tablet. The following examples are given below in order to illustrate more fully the present invention without, however, limiting the scope thereof.

EXAMPLE 1: DISINFECTANT FOR LAUNDRY

INGREDIENTS		grammes/tablet
PARAFFIN (melting point 60°C)	1	0.195
PERFUME	2	0.01
TAED	3	1.86
AEROSIL 200	4	0.146
SODIUM PERCARBONATE	5	4.16

5

ANHYDROUS SODIUM SULPHATE	6	0.25
TALC	7	0.19
PEG 6000	8	0.019
Sieved CETRIMIDE	9	0.37
TOTAL		7.2 g/tablet

Production method: Mix the perfume with the melted paraffin and mix the TAED into the mixture, cool and granulate. The granulate is mixed inside a screw mixer with the substances 4, 5, 6, 7, 8, 9 and the mixture thus obtained is compressed, forming tablets of 7.2 g.

EXAMPLE 2: DISINFECTANT TABLETS FOR DISHWASHERS

INGREDIENTS	grammes/tablet
PARAFFIN	0.3163
TAED	3.0964
AEROSIL 200	0.5327
SODIUM PERCARBONATE	5.6934
OSIS 70 (HYDROGENATED VEGETABLE FAT -	0.4794
melting point 70°C )	
ANHYDROUS SODIUM SULPHATE	0.4162
TALC	0.3163
PEG 6000	0.0333
CETRIMIDE	0.6160
TOTAL	11.5 g/ tablet

6

EXAMPLE 3: MILD BLEACH FOR WASHING MACHINES

INGREDIENTS	mg/tablet
SODIUM PERCARBONATE PEROXY HYDRATE	12149.42
IGOR 7% (HYDROGENATED PALM OIL -	850.46
melting point 60°C)	
PHENOL PHTHALEIN	0.12
TOTAL	13 g/tablet

# EXAMPLE 4: DETERGENT TABLETS

INGREDIENTS	grammes
SODIUM ALKYLBENZENE	24
SULPHONATE	
SODIUM TRIPOLYPHOSPHATE	25
SODIUM METASILICATE	4
ANHYDROUS SODIUM SULPHATE	3
CMC	10
ALKYNOLAMIDE	2
SODIUM CARBONATE	12
HYDROGENATED PALM OIL	20
(melting point 57°C/60°C)	
TOTAL	100 g (five 20 gramme
	tablets)

7

# **EXAMPLE 5: DETERGENT TABLETS**

INGREDIENTS	grammes
CRYSTALLINE SODIUM	90
CARBONATE	
ANHYDROUS SODIUM SULPHATE	10
HEXAMETAPHOSPHATE	10
SODIUM METASILICATE	20
SULPHONATED LAURYL ALCOHOL	40
SODIUM PERBORATE	10
HYDROGENATED PALM OIL	20
(melting point 65°C/70°C)	
TOTAL	200 g (ten 20 gramme
	tablets)

# EXAMPLE 6: DETERGENT TABLETS FOR DISHWASHERS

INGREDIENTS	grammes
TETRASODIUM PYROPHOSPHATE	53
ANHYDROUS SODIUM	35
METASILICATE	
TRICHLORO-ISOCYANURIC	1
ACID	
ANIONIC DETERGENT	1
PARAFFIN (melting point	10
60°C)	
TOTAL	100 g (five 20 gramme
	tablets)

8

**EXAMPLE 7: DISINFECTANT TABLETS FOR DISHWASHERS** 

INGREDIENTS	grammes/tablet
PARAFFIN (melting point 56°C /	0.3163
58°C)	
TAED	3.0964
AEROSIL 200	0.5327
SODIUM PERCARBONATE	5.6934
HYDROGENATED VEGETABLE FAT	0.4794
(melting point 59°C / 68°C)	
ANHYDROUS SODIUM SULPHATE	0.4162
TALC	0.3163
PEG 6000	0.0333
CETRIMIDE	0.6160
TOTAL	11.5 g/tablet

Tablet decomposition test performed in a temperature-regulated dissolver:

Test parameters	DISINFECTANT FOR
	DISHWASHERS
Apparatus	SOTAX dissolver
Working temperature	45° C
Speed of rotation of blades	140 RPM
Solvent medium used	Deionized water
Working conditions	One 11.5 g tablet in
	800 ml
Number of repetitions	Pr.13004/2 = 1
performed	
Results	Pr.13004/2 = 1 hour

EXAMPLE 8: MILD BLEACH FOR WASHING MACHINES

INGREDIENTS	mg/tablet
SODIUM PERCARBONATE PEROXY HYDRATE	12149.42
HYDROGENATED VEGETABLE FAT	850.46
(melting point 57°C / 60°C	
PHENOL PHTHALEIN	0.12
TOTAL	13 g/tablet

Production method: mix the percarbonate with 2% of hydrogenated vegetable fat, compress and dry-granulate the tablets obtained.

Place the granulate inside a screw mixer and coat it using liquid fat mixed with the phenol phthalein spread over the granulate whilst stirring. Cool and compress, forming 13 g tablets.

Tablet decomposition test performed in a temperature-regulated dissolver.

Test parameters:	Mild bleach for washing machines
Apparatus	SOTAX dissolver
Working temperature	Test 1 = 30° C
	Test 2 = 40° C
Speed of rotation of	140 RPM
blades	
Solvent medium used	Deionized water
Working conditions	One 14.0 g tablet in 800 ml
Number of repetitions	3
performed	
Results	30° C = more than 7 hours 30 min
	40° C = more than 7 hours 30 min

### CLAIMS

1. Controlled release detergent tablet comprising 50 to 99% by weight of at least one active component with a detergent action and 1 to 50% by weight of at least one hydrophobic substance.

- 2. Detergent tablet according to Claim 1, characterized in that said active component with a detergent action is present in an amount equal to 60-99% by weight, preferably in an amount equal to 65-95% by weight.
- 3. Tablet according to any one of the preceding claims, characterized in that said hydrophobic substance is present in an amount equal to 1-30% by weight, preferably in amount equal to 1-15% by weight.
- 4. Tablet according to any one of the preceding claims, characterized in that said hydrophobic substance has a melting point of between 30 and 80° C.
- 5. Tablet according to any one of the preceding claims, characterized in that said hydrophobic substance has a melting point of between 55 and 75° C.
- 6. Tablet according to any one of the preceding claims, characterized in that said hydrophobic substance is selected from among hydrogenated vegetable fats, waxes, animal and vegetable fats and paraffins.
- 7. Tablet according to Claim 6, characterized in that said hydrogenated vegetable fat is selected from among hydrogenated palm oil, hydrogenated castor oil and hydrogenated vegetable oils.

- 8. Tablet according to any one of the preceding claims, characterized in that said active component with a detergent action is chosen from among oxidizers, detergents and/or softener and active components which interact with the released oxygen.
- 9. Tablet according to Claim 8, characterized in that said oxidizers are chosen from among percarbonates, persulphates and perborates.
- 10. Tablet according to Claim 8, characterized in that said active component which interacts with the released oxygen is TAED.
- 11. Tablet according to Claim 8, characterized in that said detergents and/or softener are chosen from among phosphates, polyphosphates, EDTA, citric acid, sodium bicarbonate, sodium carbonate, alkyl sulphonates, trichloroisocyanuric acid, potassium dichloroisocyanurate, chloramine-T, phosphates, pyrophosphates, tripolyphosphates, potassium silicate, sodium silicate, xylene sulphonate, toluene sulphonate, cetrimide, sodium sulphonate, alkylaryl sulphonate, monoisopropanolamide, sodium metasilicate, sodium lauryl sulphate, non-ionic surfactants, anionic surfactants, cationic surfactants, protease, lipase,  $\alpha$ -amylase, βamylase, borax and sodium sulphate.
- 12. Tablet according to any one of the preceding claims, characterized in that it contains excipients in an amount of between 0.1 and 20% by weight.
- 13. Tablet according to Claim 12, characterized in that said excipients are present in an amount of between 0.5 and 15% by weight, and preferably between 1 and

10% by weight.

14. Tablet according to Claim 12, characterized in that said excipients are chosen from among carbonates, phosphates, sulphates, cellulose and cellulose derivatives, sugars, amides and proteins.

- 15. Tablet according to any one of the preceding claims, characterized in that it is in the form of a coated core, the core essentially consisting of said at least one active component with a detergent action and the coating essentially consisting of said at least one hydrophobic substance.
- 16. Tablet according to any one of the preceding claims, characterized in that it is in the form of a compressed granulate consisting essentially of a mixture of said at least one active component with a detergent action and said at least one hydrophobic substance.
- 17. Detergent tablet with two or more layers, consisting of at least one immediate release layer and at least one layer for controlled release of at least one active component with a detergent action, said controlled release layer consisting of a tablet according to any one of the preceding claims.
- 18. Use of a tablet according to any one of the preceding claims for washing fibres and/or fabrics.

# INTERNATIONAL SEARCH REPORT

International application No PCT/EP2006/050681

CLASSIFICATION OF SUBJECT MATTER VV. C11D17/00 C11D3 INV. C11D3/20 C11D3/18 C11D3/37 According to International Patent Classification (IPC) or to both national classification and IPC Minimum documentation searched (classification system followed by classification symbols) C11D Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data, PAJ C. DOCUMENTS CONSIDERED TO BE RELEVANT Category\* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. X WO 95/25161 A (HENKEL 1-6, KOMMANDITGESELLSCHAFT AUF AKTIEN; KRUSE, 8-11,16,HANS; JACOBS, JOCHEN;) 21 September 1995 (1995-09-21) Υ page 10, paragraph 3; claims 1-15; 1 - 18examples 1-11 page 2, paragraph 4 page 3, paragraph 4 - page 4, paragraph 1 page 7, paragraph 3 - page 8, paragraph 1 χ EP 0 851 024 A (UNILEVER N.V; UNILEVER 1-6,8,9,PLC) 1 July 1998 (1998-07-01) 11-14.16,17 page 3, lines 18,19,32-35; claims 1.7; 7,10 tables 1,3 page 1, paragraphs 5,6 -/-χ Further documents are listed in the continuation of Box C. See patent family annex. Special categories of cited documents: \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the \*A\* document defining the general state of the art which is not considered to be of particular relevance invention "E" earlier document but published on or after the international \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another involve an inventive step when the document is taken alone document of particular relevance; the claimed invention citation or other special reason (as specified) cannot be considered to involve an inventive step when the document is combined with one or more other such docu-"O" document referring to an oral disclosure, use, exhibition or ments, such combination being obvious to a person skilled in the art. other means document published prior to the international filing date but later than the priority date claimed \*&\* document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 11 May 2006 07/06/2006 Name and mailing address of the ISA/ Authorized officer European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Klier, E Fax: (+31-70) 340-3016

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