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(54) Title: DETERGENT COMPOSITIONS

(57) Abstract

A detergent tablet for fabric washing is compacted from a particulate composition containing 5 to 50 % detergent active, 2 to 60 % of water–soluble inorganic phosphate builder and other ingredients including 8 to 30 % of peroxygen bleach, wherein the tablet has a density of at least 1040 gm/litre and the bleach is sodium percarbonate, sodium perborate tetrahydrate or a mixture of them. This selection of peroxygen bleach leads to a better combination of strength before use with speed of disintegration at the time of use.

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DETERGENT COMPOSITIONS

This invention relates to the manufacture of detergent compositions in the form of tablets intended to be consumed when washing a single load of laundry.

When manufacturing a detergent composition for fabric washing there are a number of possible options. Such compositions have for many years been manufactured in particulate form, commonly referred to as powders. Detergent compositions can also be manufactured as liquids. Tablets, to which this invention relates, are yet another possibility.

When formulating a detergent composition there is scope to make both qualitative and quantitative choices concerning the ingredients. Anionic detergent actives are the most commonly used, usually together with nonionic detergent actives. Amongst the anionic detergent actives which are commercially available, linear alkylbenzene sulphonate and primary alkyl sulphate are commonly used. There has been a trend for particulate detergent compositions to be manufactured with a bulk density higher than 650 g/litre which is a departure from older practice when bulk densities were customarily lower.

Detergent compositions in tablet form have, potentially at least, several advantages over powder products. They do not require the user to measure out a volume of powder or liquid. Instead one or several tablets provide an appropriate quantity of composition for washing a single load in a washing machine or possibly by hand. They are thus easier for the consumer to handle and dispense.

Detergent compositions in tablet form are generally made by compressing or compacting a detergent powder which includes both detergent active and detergency builder. It is desirable that tablets have adequate strength when dry, yet disperse and dissolve quickly when added to wash water. Detergent tablets for fabric washing typically contain at least 5% by weight of detergent active. This serves as a binder, but can also retard disintegration and dissolution of a tablet. (By contrast, tablets for use in automatic dishwashing machines typically contain 2% by weight or less of detergent active, customarily a low-foaming nonionic detergent, as in WO96/23053 for instance). There have been a number of disclosures relating to the manufacture of detergent tablets for fabric washing which have both strength and rapidity of disintegration in water, for example EP-A-522766.

GB-A-1080066 teaches that tablets should have void space between particles in order to allow penetration of water into the tablet at the time of use. The teaching of this document is that the void volume should be from 35 to 60% of the total tablet volume. US-A-3081267 teaches that void space within a tablet and communicating with external air should be from 40 to 60% by volume of the tablet.

Both of these documents teach that after the tablets are made by compaction of the particulate detergent composition the tablets should be sprayed on their exterior with water, which is then allowed to dry. The effect of this is to cause partial hydration and dissolution at the exterior of the tablets thus cementing material together at the tablet exterior and enhancing tablet strength.

These documents date from 1963-1966. In more recent documents there has been disclosure of tablets of lower void volume and in Example 6 of EP 711828 tablets are disclosed which contain aluminosilicate as builder and have porosities corresponding to 30% or 20% of air in the tablet volume.

When making tablets from particulate detergent composition, it is desirable that the tablets should dissolve/disintegrate rapidly when added to water for use, yet have a good mechanical strength prior to use. These properties are antagonistic. As more pressure is used when a tablet is compacted, so the tablet density and strength rise, but the speed of disintegration/dissolution goes down.

This invention is concerned with tablets compacted from a detergent composition containing a substantial portion of water-soluble phosphate builder.

Unexpectedly, we have found that in such tablets, the combination of tablet strength and speed of disintegration is affected by the choice of material incorporated as peroxygen bleach. Sodium percarbonate and sodium perborate tetrahydrate both give properties which are better than those obtained with sodium perborate monohydrate.

Consequently, in a first aspect the present invention provides a detergent tablet compacted from a particulate composition containing:

from 2% preferably 5% up to 50% by weight of detergent active,

from 20 to 60% by weight of water-soluble inorganic phosphate builder, and

other ingredients,

wherein the tablet has a density of at least 1040 gm/litre and contains from 8 to 30% by weight of peroxygen bleach which is sodium percarbonate or sodium perborate tetrahydrate or a mixture thereof.

Sodium percarbonate has been found to give even better strength than perborate tetrahydrate and is therefore preferred.

The amount of peroxygen bleach is preferably at least 10% by weight of the composition. The amount may be not more than 25% or even 20% by weight.

The peroxygen bleach is preferably distributed throughout the tablet, although it may be present as a multiplicity of granules distributed throughout the tablet. Notably, sodium percarbonate may be utilised in the form of granules with a water-soluble coating of a protective material serving to keep moisture away from the percarbonate until the time of use.

Tablet density preferably lies in a range from 1040 or 1050gm/litre up to 1300gm/litre. The tablet density may well lie in a range up to 1250 or even 1200gm/litre.

Tablet density is inversely related to tablet porosity, which is conveniently expressed as the percentage of its volume which is air (i.e. empty space).

The air content of a tablet can be calculated from the volume and weight of the tablet, provided the true density of the solid content is known. The latter can be measured by compressing a sample of the material under vacuum with a very high applied force, then measuring the weight and volume of the resulting solid object.

The true density of a detergent composition is often about

1.6, in which case a tablet density range from 1040 to 1300g/litre is approximately 19 to 35% air by volume. Ranges of 1040 to 1250 or 1200g/litre approximates to 22 or 25% to 35% air by volume. A preferred range of 1070 to 1250g/litre approximates to 22 to 33% air by volume.

Preferably, part or all of the detergent active is anionic. We have found that satisfactory strength and speed of disintegration can be obtained using a detergent active mixture in which anionic and nonionic detergent are in proportions from 1.5:1 to 4:1, better 1.8:1 to 3:1 or 4:1. These proportions give good cleaning. Therefore, in a second aspect the invention provides a detergent tablet compacted from a particulate composition containing:

from 2% preferably at least 5% up to 50% by weight of detergent active,

from 20 to 60% by weight of water-soluble inorganic phosphate builder, and

other ingredients,

wherein the tablet has a density of at least 1040gm/litre and the detergent active contains anionic and nonionic detergent in proportions of 1.5:1 to 4:1.

It is preferred that the nonionic detergent active is a mixture of an ethoxylated fatty alcohol with HLB value over 11.0 and an ethoxylated fatty alcohol with HLB value below 9.5 better below 9.0.

The particulate composition which is compacted may be a mixture of particles of individual ingredients, but usually will comprise particles which themselves contain a mixture of ingredients. Such particles containing a mixture of ingredients may be produced by a granulation process and may be used alone or together with particles or single ingredients.

The composition will contain detergent active and detergent builder. Other ingredients are optional, but usually there will be some other ingredients in addition to the detergent active and detergency builder.

The amount of detergent active in a tablet is suitably from 5 or 8wt% up to 40 to 50wt%. Detergent-active material present may be anionic (soap or non-soap), cationic, zwitterionic, amphoteric, nonionic or any combination of these.

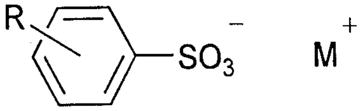
Anionic detergent-active compounds may be present in an amount of from 0.5 to 40 wt%, preferably from 2%, 4% or 5% up to 30 or 40 wt%.

Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

Primary alkyl sulphate having the formula

$$ROSO_3^- \ M^+$$

in which R is an alkyl or alkenyl chain of 8 to 18 carbon atoms especially 10 to 14 carbon atoms and M^+ is a solubilising cation especially sodium, is commercially significant as an anionic detergent active. Linear alkyl benzene sulphonate of the formula



where R is linear alkyl of 8 to 15 carbon atoms and M^+ is a solubilising cation, especially sodium, is also a commercially significant anionic detergent active.

Frequently, such linear alkyl benzene sulphonate or primary alkyl sulphate of the formula above, or a mixture thereof will be the desired anionic detergent and may provide 75 to 100wt% of any anionic non-soap detergent in the composition.

In some forms of this invention, the amount of non-soap anionic detergent lies in a range from 5 to 30 wt% of the composition, better 5 to 20 wt%.

It may also be desirable to include one of 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.

Suitable nonionic detergent 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 either alone or with propylene oxide.

Specific nonionic detergent compounds are alkyl (C_{8-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols with ethylene oxide, copolymers of ethylene oxide and propylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-diamine.

Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{10-15} primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

In certain forms of this invention the amount of nonionic detergent lies in a range from 1 to 20%, better 2% up to 15% or 20% by weight of the composition.

Preferred is to use from 5 to 20% of non-soap anionic detergent, especially linear alkyl benzene sulphonate or primary alkyl sulphate, together with 2 to 15% of nonionic detergent, especially ethoxylated fatty alcohol, where the ratio of anionic to nonionic is in the range from 1.5:1 to 4:1.

Products of this invention also include phosphate detergency builder, which may be an alkali metal orthophosphate, pyrophosphate or tripolyphosphate. Preferred is sodium tripolyphosphate.

Examples of other water-soluble builders which may be present are carbonates, e.g. sodium carbonate; and organic builders containing up to six carbon atoms, e.g. sodium tartrate, sodium citrate, trisodium carboxymethyloxysuccinate.

The amount of phosphate or polyphosphate detergency builder is at least 20% by weight, often at least 26% or even at least 33% by weight of the overall composition.

The total amount of detergency builder will generally lie in a range from 5 to 80wt% of the composition. The amount may be at least 10 or 15wt% and may lie in a range up to 50 or 60wt%.

The sodium perborate tetrahydrate or sodium percarbonate is advantageously employed together with an activator. Bleach activators, also referred to as bleach precursors, have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example, tetraacetylethylene diamine (TAED), now in widespread commercial use in conjunction with sodium perborate; and perbenzoic acid precursors. Typically activator used as 1 to 10% by weight of a composition.

Other ingredients may also be present in the overall composition. These include sodium carboxymethyl cellulose, colouring materials, enzymes, fluorescent brighteners, germicides, perfumes and bleaches. Sodium alkaline silicate may be included, although the amount of this or at least the amount added as an aqueous liquid, is preferably restricted so as to keep to a particulate mixture prior to compaction.

The detergent composition may incorporate a binder which is water-soluble and serves as a disintegrant by disrupting the structure of the tablet when the tablet is immersed in water, as taught in our EP-A-522766.

Such a binder material should melt at a temperature of 35°C, better 40°C or above, which is above ambient temperatures in many temperate countries. For use in hotter countries it will be preferable that the melting temperature is somewhat above 40°C, so as to be above the ambient temperature.

For convenience the melting temperature of the binder material should be below 80°C.

Preferred binder materials are synthetic organic polymers of appropriate melting temperature, especially polyethylene glycol. Polyethylene glycol of average molecular weight 1500 (PEG 1500) melts at 45°C and has proved suitable. Polyethylene glycols of molecular weight 4000 and 6000 melt at about 55°C and 62°C respectively.

Other possibilities are polyvinylpyrrolidone, and polyacrylates and water-soluble acrylate copolymers.

We have found it desirable that the particulate composition which is compacted should have a bulk density of at least 650 g/litre, preferably at least 700 g/litre, and advantageously at least 750 g/litre.

Granular detergent compositions of high bulk density can be prepared by granulation and densification in a high-speed mixer/granulator, as described and claimed in EP 340013A (Unilever), EP 352135A (Unilever), and EP 425277A (Unilever), or by the continuous granulation/densification processes described and claimed in EP 367339A (Unilever) and EP 390251A (Unilever).

The invention can be put into effect using a conventional stamping press. A suitable press will generally have a pair of mould parts which move relatively towards and away from each other to compact particulate material between them. They may move within a surrounding sleeve or similar structure.

For any chosen composition, the density and strength of tablets varies with the pressure applied to compact the composition into tablets.

The amount of pressure needed to obtain a density in the required range can be found by making tablets with varying amounts of applied force, and determining the density of the tablets obtained.

The tablets may be made without any spray on of water after stamping the tablets.

Example 1

Tablets for use in fabric washing were made, starting with a base powder of the following composition:

	parts by weight
Sodium linear alkylbenzene sulphonate	9.62
C_{13-15} fatty alcohol 7EO	1.07
C_{13-15} fatty alcohol 3EO	3.21
Soap	0.27
Sodium tripolyphosphate, type 1A [1]	24.31
Sodium silicate	5.88
Sodium carboxymethyl cellulose	0.21
Acrylate/maleate copolymer	1.15
Salts, moisture and minor ingredients	8.9
	54.60

1. This tripolyphosphate contained less than 30% of the phase I form of anhydrous sodium tripolyphosphate.

Three powders were made by mixing this base powder with persalts, sodium tripolyphosphate specified to contain 70% phase I form and contain 3.5% water of hydration (Rhodia-Phos HPA 3.5 available from Rhone-Poulenc) and other detergent ingredients as tabulated below.

The compositions thus contained the following percentages by weight:

	% by weight		
	A	B	C
Base powder	54.60	54.60	54.60
HPA sodium tripolyphosphate	21.22	21.22	20.12
TAED granules	3.35	3.35	3.35
Sodium carbonate	3.20	4.95	--
Sodium percarbonate	15.00	--	--
Sodium perborate monohydrate	--	13.25	--
Sodium perborate tetrahydrate	--	--	19.30
Anti-foam granule	1.16	1.16	1.16
Enzymes, phosphonate, perfume	1.47	1.47	1.47

The amounts of persalt were chosen to give the same amounts of available oxygen. The sodium percarbonate was in the form of granules with a water-soluble coating.

35g portions of each composition were made into cylindrical tablets of 44 mm diameter, using a Carver hand press. The force applied to make the tablets was the same (300kg) in each case.

The tablets were weighed and measured to determine their density which was found to be 1117gm/litre ± 5% in each case.

The strength of the tablets was determined by the following test of their diametral fracture stress. The test procedure was carried out using a testing machine with flat faces which were urged together by a measured force, such as an Instron Universal Testing Machine.

The cylindrical tablet was placed between the platens of an Instron machine, so that the platens contact the curved surface of the cylinder at either end of a diameter through the tablet. The sample tablet was then compressed diametrically, by advancing the platens of the machine towards each other at a slow rate such as 1cm/min until fracture of the tablet occurred, at which point the applied load required to cause fracture was recorded. The diametral fracture stress was then calculated from the following equation:

$$\delta_O = \frac{2P}{\pi Dt}$$

where δ_O is the diametral fracture stress in Pascal (Pa), P is the applied load in Newtons (N) to cause fracture, D is the tablet diameter in metres (M) and t is the tablet thickness, also in metres (M).

We have found it desirable in this invention that tablets should have a DFS of at least 6KPa, better at least 8KPa. DFS will usually not need to exceed 40KPa, and a range from 10 to 40KPa is particularly preferred. Values of DFS up to at least 60KPa may be used, however.

The break-up, dispersion and dissolution of tablets was measured by a test procedure in which a tablet is placed on a plastic sieve with 2mm mesh size which was immersed in 9 litres of demineralised water at ambient temperature of 22°C and rotated at 200 rpm. The water conductivity was monitored until it reached a constant value.

The time for break up and dispersion of the tablets was taken as the time (T_{90}) for change in the water conductivity to reach 90% of its final magnitude. This was also confirmed by visual observation of the material remaining on the rotating sieve.

The results obtained were:

	A percarbonate	**B** perborate mono-hydrate	**C** perborate tetra-hydrate
Tablet strength (kPa)	32	20	27
Tablet dissolution T_{90} (min)	3.5	5.2	3.8

This shows that tablets made with percarbonate and with perborate tetrahydrate had unexpectedly greater strength than tablets made with perborate monohydrate. They also dissolved more quickly, in spite of their greater strength.

CLAIMS:

1. A detergent tablet compacted from a particulate composition containing:

from 5 to 50% by weight of detergent active,

from 20 to 60% by weight of water-soluble inorganic phosphate builder, and

other ingredients,

wherein the tablet has a density of at least 1040 gm/litre and contains from 8 to 30% by weight of peroxygen bleach which is sodium percarbonate or sodium perborate tetrahydrate or a mixture thereof.

2. A tablet according to claim 1 wherein the peroxygen bleach is sodium percarbonate.

3. A tablet according to claim 1 or claim 2 containing from 30 to 60% by weight of sodium tripolyphosphate as the water-soluble inorganic phosphate builder.

4. A tablet according to any one of claims 1 to 3 which contains at least 4% by weight of anionic detergent and contains in total from 5 to 40% by weight of detergent active.

5. A tablet according to any one of claims 1 to 4 which contains from 5 to 30% by weight of anionic detergent.

6. A tablet according to claim 5, which contains from 2 to 20% by weight of nonionic detergent.

7. A tablet according to claim 4, claim 5, or claim 6 which contains from 5 to 30% by weight of anionic detergent and from 2 to 20% by weight of nonionic detergent.

8. A tablet according to any one of claims 1 to 7 wherein the detergent active contains anionic and nonionic detergent active in weight ratio of 1.5:1 to 4:1.

9. A tablet according to claim 6, claim 7, or claim 8 wherein the nonionic detergent is a mixture of a nonionic detergent with HLB value over 11.0 and a nonionic detergent with HLB value below 9.5, in a weight ratio lying in a range from 3:1 to 1:3.

10. A tablet according to any one of claims 1 to 9 having density in the range from 1075 to 1275 gm/litre.

11. A tablet according to any one of claims 1 to 10 having porosity in the range from 19 to 35% air by volume.

12. A tablet according to any one of claims 1 to 10 having porosity in the range from 22 to 33% air by volume.

INTERNATIONAL SEARCH REPORT

Int. .ional Application No
PCT/EP 98/01769

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D17/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 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)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 711 828 A (UNILEVER PLC) 15 May 1996 cited in the application see examples ---	1,2,4-12
A	GB 911 204 A (UNILEVER LTD.) 21 November 1962 see the whole document ---	1-7
A	US 3 557 003 A (R. L. MORRIS) 19 January 1971 see claims; examples ---	1-11
A	US 3 081 267 A (R. P. LASKEY) 12 March 1963 cited in the application see claims --- -/--	1,3,4, 11,12

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search 30 June 1998	Date of mailing of the international search report 09/07/1998
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
A	GB 1 080 066 A (THE PROCTER & GAMBLE COMPANY) 9 March 1966 cited in the application see page 3, column 2, line 105 - line 129 see claim 1 ---	1,3-8,11
A	US 3 953 350 A (FUJINO ET. AL.) 27 April 1976 see the whole document ---	1-5
A	WO 96 23053 A (HENKEL KG AA) 1 August 1996 cited in the application see examples; tables -----	1-9

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Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

Int .ional Application No
PCT/EP 98/01769

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