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(54) Title: DETERGENT COMPOSITION

(57) Abstract: A particulate detergent composition which comprises: (i) from 0 to 30 wt% of base powder particles, (ii) from 10 to 90 wt% of high active granules comprising at least 40 wt% non-soap surfactant, and (iii) from 10 to 90 wt% of particulate water-soluble builder. Also use of the composition in a washing process.

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DETERGENT COMPOSITION

TECHNICAL FIELD

The present invention relates to a particulate laundry detergent composition comprising a blend of different ingredients.

BACKGROUND AND PRIOR ART

The prior art is replete with examples of particulate laundry detergent compositions which have been proposed to provide improved dissolution in wash water.

As a potential solution to this problem a number of patents have suggested the use of the so-called 'adjunct' approach. This involves the segregation of negatively interacting materials. The known approaches use a high amount of base powder.

WO 96/06916 (Unilever) discloses a detergent composition which comprises a high active adjunct in combination with a base powder and other minor ingredients.

WO 98/54286 (Unilever) discloses a detergent composition comprising a granular component having a surfactant level of at least 60 wt% in combination with a base powder.

WO 98/54287 (Unilever) discloses a detergent composition comprising a granular component having a surfactant level of at least 60 wt% in combination with a base powder.

It is therefore an object of the present invention to provide a detergent composition with improved dissolution properties.

The present inventors have found that by using less base powder together with a high surfactant concentration adjunct, a detergent composition is provided with particularly good dissolution properties.

Accordingly the present invention provides a detergent composition according to claim 1 and to the use of that composition for machine washing, especially of fabric articles.

DETAILED DESCRIPTION OF INVENTION

The Composition

The particulate detergent composition of the present invention comprises two essential elements. The first element is the presence of a high active non-soap surfactant granule. The second element is the present of water soluble builder particles. Thus the particles of the high active granules are distinct from the particles of builder and are not agglomerated together to form a homogenous blend. In this way the advantages of the invention are realised.

The composition thus comprises from 10 to 90 wt% of high active granules. Preferably is comprises from 20 to 80 wt%, more preferably from 30 to 70 wt%, most preferably from 40 to 60 wt% of high active granule. Use of high active

granules at a level above 40 wt% and less than 80 wt% is desirable.

The composition thus comprises from 10 to 90 wt% of water soluble builder particles. Preferably is comprises from 20 to 80 wt%, more preferably from 30 to 70 wt%, most preferably from 40 to 60 wt% of water soluble builder particles.

The bulk density can be in the range of from 300 to 1500 g/l, depending on the composition and the method of manufacture. It is preferred, however, that the composition be of a high bulk density and is in the region of from 600 to 1500 g/l.

The High Active Granule

More than one type of high-active granules (ii) may he present, provided that in total at least 10% by weight of the composition is constituted by these granules.

It is particularly preferred that the high-active granules (ii) containing at least 40% by weight of non-soap surfactant comprise a substantial quantity of anionic surfactant.

Preferably, the granules comprise at least 50 wt%, more preferably at least 55 wt%, even more preferably at least 60 wt%, and most preferably at least 70 wt% by weight of non-soap surfactant. The particle size of the high active granule is desirably less than 700 micron.

A method of producing a detergent component containing at least 40% by weight of anionic surfactant is set forth in WO 97/32002A (Unilever). The process comprises the steps of feeding a paste material comprising water and an anionic surfactant into a drying zone, heating the paste material in the drying zone to reduce the water content thereof and subsequently cooling the paste material in a cooling zone to form detergent particles, characterised by introducing a layering agent into the cooling zone during the cooling step. This process may be carried out in a machine manufactured by VRV Impianti SpA, having a heating surface area of 1.2 m^2. The heating zones are maintained at a temperature in the region of 120-1900C, for example 170°C.

Cooling is achieved using ambient process water at 15'C. The apparatus is used with tip speed of the blades of 30 m/s.

A method of producing a detergent component containing at least 75% by weight of anionic surfactant is set forth in WO 96/06916A and WO 96/06917A (Unilever). In the process of the former application, a paste material comprising water in an amount of more than 10% by weight of the paste and the surfactant is fed into a drying zone, the paste is heated to a temperature in excess of 1300C to reduce the water content to more than 10% by weight and the material is subsequently cooled to form detergent particles.

It is highly preferred that the non-soap surfactant in the high active granules comprises anionic surfactant.

Preferably, a major proportion of the anionic surfactant is contained in the high-active granules (ii) containing at least 40% by weight of anionic surfactant. The remainder of the anionic surfactant may be present in any other suitable form. Possibly, the remainder of the anionic surfactant will be present in the base powder if any is present. Preferably, at least 60% by weight of the anionic surfactant is incorporated in the high-active granules. Most preferably the high-active granule is silicate free due to the difficulty of making granules containing silicate using the preferred processes.

Nonionic surfactant may also be present in the form of high-active granules, although these need not necessarily contain at least 60% by weight of nonionic surfactant. A method of manufacturing nonionic surfactant containing granules comprising 55% by weight or more of nonionic surfactant, at least 5% by weight of silica of oil absorption capacity of 1.0 ml/g and less than 10% by weight of aluminosilicate are disclosed in WO98/54281. These granules can be manufactured by mixing together components in a high speed granulator (for example an Eirich R502 Granulator). Alternatively, 70 to 100% by weight of the solid components and 70 to 95% by weight of the nonionic surfactant can be mixed together in a first step, the remainder of the solid components and nonionic surfactant being added in a second step, preferably under moderate shear. In the second process, the majority of the structurant is preferably added in the second step.

Organic Detergent Surfactant

Finished detergent compositions of the present invention contain an organic detergent surfactant. Components for detergent compositions which are also part of the invention do not necessarily comprise surfactant.

Detergent-active compounds (surfactants) may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch. The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds. The total amount of surfactant present is suitably within the range of from 5 to 60 wt%, preferably from 5 to 40 wt%.

Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; primary and secondary alkylsulphates, particularly C_8-C_{20} primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C_8-C_{20}

aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C_{10}-C_{15} primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkanolamides, alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

Cationic surfactants that may be used include quaternary ammonium salts of the general formula $R_1R_2R_3R_4N^+ X^-$ wherein the R groups are long or short hydrocarbyl chains, typically alkyl, hydroxyalkyl or ethoxylated alkyl groups, and X is a solubilising anion (for example, compounds in which R_1 is a C_8-C_{22} alkyl group, preferably a C_8-C_{10} or C_{12}-C_{14} alkyl group, R_2 is a methyl group, and R_3 and R_4, which may be the same or different, are methyl or hydroxyethyl groups); and cationic esters (for example, choline esters).

Amphoteric and zwitterionic surfactants that may be used include alkyl amine oxides, betaines and sulphobetaines. In accordance with the present invention, the detergent surfactant (a) most preferably comprises an anionic sulphonate or sulphonate surfactant optionally in admixture with one or more cosurfactants selected from ethoxylated nonionic surfactants, non-ethoxylated nonionic surfactants, ethoxylated sulphate anionic surfactants, cationic surfactants, amine oxides, alkanolamides and combinations thereof.

Surfactants are preferably present in a total amount of from 5 to 60 wt%, more preferably from 10 to 40 wt%.

Base Powder

Base powders conventionally form the 'base' of a detergent composition onto which further additional ingredients are added. They are therefore a useful way of providing a 'core' of ingredients which are common to a wide range of detergent ingredients. For the purposes of the present invention such base powders contain surfactant and builder. However, the present invention does not require a large amount of base powder in order to provide the advantages of the improved physical properties. As a result it is a requirement that the detergent composition of the present invention comprise from 0 to 30 wt% of base powder, preferably from 0 to 20 wt% and preferably from 0 to 10 wt%. The composition could therefore be free of base powder.

It is a characteristic of base powders that the ingredients they contain are homogeneously distributed within. In contrast, the ingredients of the present invention are not homogeneous and are usually simply dry-mixed together.

Base powders of low to moderate bulk density may be prepared by spray-drying a slurry, and optionally postdosing (dry-mixing) further ingredients. "Concentrated" or "compact" powders may be prepared by mixing and granulating processes, for example, using a high-speed mixer/granulator, or other non-tower processes.

Water Soluble Builder

Finished detergent compositions of the present invention contain water-soluble builder.

The compositions of the invention suitably contain from 10 to 80%, preferably from 15 to 70% by weight, of detergency builder. Preferably, the quantity of builder is in the range of from 15 to 50% by weight.

Preferably the builder is selected from sodium tripolyphosphate, sodium carbonate, sodium citrate and combinations of these.

Preferred builders according to the present invention are phosphate builders, especially sodium tripolyphosphate. This may be used in combination with sodium orthophosphate, and/or sodium pyrophosphate.

Other inorganic builders that may be present additionally or alternatively include sodium carbonate, layered silicate, amorphous aluminosilicates, however, layered silicates may not be used as a soluble builder component.

Organic builders that may be present include polycarboxylate polymers such as polyacrylates and acrylic/maleic copolymers; polyaspartates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-di- and trisuccinates, carboxymethyloxysuccinates, carboxy-methyloxymalonates, dipicolinates, hydroxyethyl-

iminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts.

Organic builders may be used in minor amounts as supplements to inorganic builders such as phosphates and zeolites. Especially preferred supplementary organic builders are citrates, suitably used in amounts of from 5 to 30 wt %, preferably from 10 to 25 wt %; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt %, preferably from 1 to 10 wt%. Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form.

Other Detergent Ingredients

As well as the surfactants and builders discussed above, the compositions may optionally contain bleaching components and other active ingredients to enhance performance and properties.

These optional ingredients may include, but are not limited to, any one or more of the following: soap, peroxyacid and persalt bleaches, bleach activators, sequestrants, cellulose ethers and esters, other antiredeposition agents, sodium sulphate, sodium silicate, sodium chloride, calcium chloride, sodium bicarbonate, other inorganic salts, proteases, lipases, cellulases, amylases, other detergent enzymes, fluorescers, photobleaches, polyvinyl pyrrolidone, other dye transfer inhibiting polymers, foam controllers, foam boosters, acrylic and acrylic/maleic polymers, citric

acid, soil release polymers, fabric conditioning compounds, coloured speckles, and perfume.

Detergent compositions according to the invention may suitably contain a bleach system. The bleach system is preferably based on peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, capable of yielding hydrogen peroxide in aqueous solution. Suitable peroxy bleach compounds include organic peroxides such as urea peroxide, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulphates. Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate. Especially preferred is sodium percarbonate having a protective coating against destabilisation by moisture. Sodium percarbonate having a protective coating comprising sodium metaborate and sodium silicate is disclosed in GB 2 123 044B (Kao).

The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%.

Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid

precursors. An especially preferred bleach precursor suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED). Also of interest are peroxybenzoic acid precursors, in particular, N,N,N-trimethylammonium toluoyloxy benzene sulphonate.

A bleach stabiliser (heavy metal sequestrant) may also be present. Suitable bleach stabilisers include ethylenediamine tetraacetate (EDTA) and the polyphosphonates such as Dequest (trademark), EDTMP.

Although, as previously indicated, in one preferred embodiment of the invention enzymes are preferably absent, in other embodiments detergent enzymes may be present. Suitable enzymes include the proteases, amylases, cellulases, oxidases, peroxidases and lipases usable for incorporation in detergent compositions.

In particulate detergent compositions, detergency enzymes are commonly employed in granular form in amounts of from about 0.1 to about 3.0 wt%. However, any suitable physical form of enzyme may be used in any effective amount.

Antiredeposition agents, for example cellulose esters and ethers, for example sodium carboxymethyl cellulose, may also be present.

The compositions may also contain soil release polymers, for example sulphonated and unsulphonated PET/POET polymers, both end-capped and non-end-capped, and polyethylene glycol/polyvinyl alcohol graft copolymers such as Sokolan

(trademark) HP22. Especially preferred soil release polymers are the sulphonated non-end-capped polyesters described and claimed in WO 95 32997A (Rhodia Chimie).

EXAMPLES

Example 1

The following high active granule was made according to the process described in WO 96/06916:

Ingredient E.g. 1 HAG	**Wt%**
Sodium C12-C18 benzene sulphonate	60
Sodium carbonate	30
Zeolite	10

These surfactant granules were dry-mixed with water soluble builder particles to produce a formulation shown in table 2 as example 1. The composition had a bulk density of around 700g/l and had excellent dissolution properties.

For comparison purposes the same formulation was made up by using a spray-dried base powder containing surfactant and builder, which was topped up to make the same formulation, indicated as comparative example A in table 2.

Example 2

A similar formulation was made from a High active granule made in the same way as for example 1 but with the composition:

Ingredient example 2 HAG	**Wt%**
Sodium C_{12}-C_{18} benzene sulphonate	65
Sodium carbonate	12
Zeolite	18
sodium sulphate	2
water and minors	3

This too was dry mixed with water soluble builder particles to produce a formulation shown in table 2 as example 2. Unlike for example 1, this formulation also had dry mixed base powder added. The ratio used was 70% HAG, 20% water-soluble sodium carbonate builder granule and 10% base powder. The composition so formed had a bulk density of about 700g/l and excellent dissolution properties. The composition of the base powder used included some further anionic surfactant and sodium carbonate, in addition to sodium silicate and sodium sulphate and various minor components normally found in a base powder.

Table 2 shows the dramatic improvement in dissolution rate when the base powder is removed and a high active granule is used (comparative example A versus example 1).

The dissolution rate is given as a T_{90} value, which is the time taken for the conductivity of a water bath containing the powder to reach 90% of its final value.

Table 2

Ingredient	Example		
	A	1	2
Anionic surfactant	22.00	35.51	46.9
STTP builder	12.96	20.92	-
Zeolite 4A	-	10.25	12.6
SCMC	0.36	0.58	0.03
Fluorescer	0.08	0.13	-
Enzymes	0.5	0.8	-
Sodium Carbonate	12.74	20.56	31.3
Sodium Silicate	6.28		0.92
Sodium Sulphate	35.27	3.32	5.5
Moisture, perfume, colourants and others	9.81	7.94	2.75
TOTAL	100.00	100.00	100.00
Dissolution rate (T_{90})	22s	14s	-

T_{90} Test Method

The change in conductivity of a volume of water is timed as the powder is dissolved in it.

Apparatus

Conductivity meter with electrode (note 1).
2 litre beaker (squat type).
Magnetic stirrer with rod (60 mm) (stirring speed 4 cm vortex)

Timer clock.
Recorder (paper speed 10 mm/min)
Thermometer (temp 20°C)

Procedure

Weigh to the nearest 0.05 g, 2.5 g of a representative sample into a 50ml beaker. Pour 1 litre of demineralised water at 20°C into a 2 litre beaker, stir with a vortex of approx. 4 cm. Put the electrode into the water and start the recorder. Pour all of the sample into the water at once. Stop the test when the recorder pen is at its maximum for 2 minutes

Calculation

Mark on the chart paper the place where the pen is at its maximum (Y_{100}). Measure the distance between the start (Y_0) and the maximum (Y_{100}) on the x-axis and calculate the time taken for complete dissolution (10 mm = 1 minute). Mark the points where 90% of the sample is dissolved ($Y_{90} = Y_{100} \times 0.90$) and repeat for 95% ($Y_{95} = Y_{100} \times 0.95$). In both instances read from (Y_0). Calculate the time taken to reach each point (10 mm = 1 minute)

Dissolution rate is given as the time it takes for 90% dissolved.

CLAIMS

1. A particulate detergent composition which comprises:
   (i) from 0 to 30 wt% of base powder particles,
   (ii) from 10 to 90 wt% of high active granules comprising at least 40 wt% non-soap surfactant, and
   (iii) from 10 to 90 wt% of particulate water-soluble builder.

2. A composition as claimed in claim 1, which comprises from 0 to 20 wt% base powder.

3. A composition as claimed in claim 1, which comprises from 0 to 10 wt% base powder.

4. A composition as claimed in any preceding claim, which comprises from 20 to 80 wt% of the high active granules.

5. A composition as claimed in any preceding claim, which comprises from 30 to 70 wt% of the high active granules.

6. A composition as claimed in any preceding claim, which comprises from 20 to 80 wt% of the water-soluble builder.

7. A composition as claimed in any preceding claim, which comprises from 30 to 70 wt% of the water-soluble builder.

8. A composition as claimed in any preceding claim, wherein the high active granules comprises at least 50 wt%, preferably at least 55wt%, non-soap surfactant.

9. A composition as claimed in claim 8, wherein the high active granules comprise at least 60 wt% non-soap surfactant.

10. A composition as claimed in any preceding claim, wherein the non-soap surfactant in the high active granule comprises anionic surfactant.

11. A composition as claimed in any preceding claim, wherein the total of (ii) and (iii) is at least 50 wt% of the composition.

12. Use of a composition according to any preceding claim in a washing process, preferably a machine laundry washing process.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C11D17/06

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 7 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

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X	WO 00/31222 A (UNILEVER PLC; UNILEVER NV; HINDUSTAN LEVER LIMITED) 2 June 2000 (2000-06-02) page 1, line 8 - line 17; claims; examples 17,20	1-12
X	EP 0 220 024 A (THE PROCTER & GAMBLE COMPANY) 29 April 1987 (1987-04-29) page 1, line 4 - line 24; claims; example 1	1-12
X	WO 98/54289 A (UNILEVER PLC; UNILEVER N.V) 3 December 1998 (1998-12-03) claims; example 3	1-12
X	WO 98/54281 A (UNILEVER PLC; UNILEVER N.V) 3 December 1998 (1998-12-03) claims; examples 8,16,17	1-12

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
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"&" document member of the same patent family

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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.
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X	WO 98/54286 A (UNILEVER PLC; UNILEVER N.V) 3 December 1998 (1998-12-03) claims; examples 1-3,11	1-12
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Information on patent family members

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