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- <sup>54</sup> Granular detergent composition.
- © A granular detergent composition is disclosed comprising a surfactant, soluble encapsulates of perfume and a detergency builder comprising zeolite P having a silicon to aluminium ratio not greater than I.33, preferably not greater than I.07, (zeolite MAP).

The composition preferably has a bulk density of at least 650g/l and may optionally contain percarbonate.

Use of zeolite MAP as detergency builder reduces perfume leakage which is a problem associated with conventional detergent compositions containing perfume capsules.

The present invention relates to a granular detergent composition and, in particular, to improvements in the detergency performance of laundry detergent compositions comprising zeolites as sequestering agents for water hardness.

Detergent compositions for heavy-duty fabric washing conventionally contain detergency builders which lower the concentration of calcium and magnesium water hardness ions in the wash liquor and thereby provide good detergency effect in both hard and soft water.

Conventionally, inorganic phosphates, such as sodium tripolyphosphate, have been used as builders for laundry detergents. More recently, alkali metal aluminosilicate ion-exchangers, particularly crystalline sodium aluminosilicate zeolite A, have been proposed as replacements for the inorganic phosphates.

For example, EP 21 491A (Procter & Gamble) discloses detergent compositions containing a building system which includes zeolite A, X or P (B) or a mixture thereof. EP 384070A (Unilever) discloses specific zeolite P materials having an especially low silicon to aluminium ratio not greater than 1.33 (hereinafter referred to as zeolite MAP) and describes its use as a detergency builder. To date, however, zeolite A is the preferred aluminosilicate detergency builder in commercially available products.

However, it has been found that there are problems associated with the use of conventional detergency builders including aluminosilicates such as zeolite A. One problem is evident in granular detergent products, especially compact products, incorporating a perfume.

Perfumes are commonly employed in detergent compositions to deliver a pleasant odour on detergent bases and on fabrics or dishes during and after a wash treatment. It is known to use soluble encapsulates of perfume incorporated in a granular detergent composition to increase the perfume delivery through the wash and on fabrics without increasing the odour impact on product beyond the threshold of consumer acceptance. A further advantage of soluble capsules is that they permit reduced perfume loses on storage as compared with sprayed on perfumes, particularly in high density detergents which have a low porosity and hence are poor substrates for sprayed on perfumes. Soluble perfume capsules also have a particular advantage in detergent compositions containing percarbonate which are poorer substrates than perborate-based compositions for retaining sprayed on perfume.

However, perfume capsules incorporated in detergent compositions have a tendency to leak, particularly if stored in high temperature and/or high moisture conditions. As a result the odour impact on the product is increased beyond consumer acceptance.

We have found that this problem can be obviated by using, as the detergency builder, zeolite MAP.

Thus, the present invention provides a granular detergent composition comprising:

- (a) a surfactant selected from anionic, nonionic, cationic, amphoteric and zwitterionic detergent-active compounds and mixtures thereof;
- (b) a detergency builder comprising zeolite P having a silicon to aluminium ratio not greater than 1.33 (zeolite MAP); and
- (c) soluble encapsulates of perfume.

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According to the present invention zeolite MAP may be the sole detergency builder or it may be employed together with a co-builder known in the art. If zeolite A is employed as cobuilder the formulation should contain preferably not more than 2% by weight zeolite A or not more than 6% by weight zeolite A if the composition is overdried, i.e. has a moisture level below 12% by weight.

The detergent composition according to the invention contains, as an essential ingredient, one or more surfactants selected from anionic, nonionic, cationic, amphoteric and zwitterionic detergent-active compounds and mixtures thereof. Such surfactants are well known and described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II by Schwartz, Perry and Berch.

Examples of suitable anionic surfactants include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of  $C_8$ - $C_{15}$ ;  $C_{12}$ - $C_{15}$  primary alkyl sulphates and their ethoxylated analogues containing from 0.25 to 6 moles of ethylene oxide per mole of alkyl sulphate; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

Examples of suitable nonionic surfactants include alkoxylated adducts of fatty alcohols containing an average of less than 5 alkylene oxide groups per molecule, for example less than 4 alkylene oxide groups per molecule e.g. 3.5 and usefully 3 alkylene oxide groups per molecule or less and usefully also greater than 0.5, or I, or 2 alkylene oxide groups per molecule.

Alkylene oxide adducts of fatty alcohols are useful as hydrophobic alkoxylated nonionic surfactants for incorporation in the detergent composition of the present invention. Suitable alkylene oxide adducts of fatty alcohols can suitably be chosen from those of the general formula:

 $R-O-(C_nH_{2n}O)_vH$ 

wherein R is an alkyl or alkenyl group having at least 10 carbon atoms, most preferably from 10 to 22 carbon atoms, y is preferably from about 0.5 to about 3.5 and n is 2 or 3.

Preferred nonionic surfactants include primary  $C_{11}$ - $C_{15}$  aliphatic alcohols condensed with an average of no more than five ethylene oxide groups per mole of alcohol, having an ethylene oxide content of less than 50% by weight.

A particularly preferred aliphatic alcohol ethoxylate is a primary alcohol having an average of 12 to 15 carbon atoms in the alkyl chain condensed with an average of three ethoxy groups per mole of alcohol.

Specific examples of suitable alkoxylated adducts of fatty alcohols are Synperonic A3 (ex ICI), which is a  $C_{13}$ - $C_{15}$  alcohol with about three ethylene oxide groups per molecule and Empilan KB3 (ex Marchon), which is lauric alcohol 3EO.

Another class of nonionic surfactants comprises alkyl polyglucoside compounds of general formula

 $RO(C_nH_{2n}O)_tZ_x$ 

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wherein Z is a moiety derived from glucose; R is a saturated hydrophobic alkyl group that contains from 12 to 18 carbon atoms; t is from 0 to 10 and n is 2 or 3; x is from 1.1 to 4, the compounds including less than 10% unreacted fatty alcohol and less than 50% short chain alkyl polyglucosides. Compounds of this type and their use in detergent compositions are disclosed in EP-B 0070074, 0070077, 0075996 and 0094118.

Where the composition comprises an aliphatic alcohol ethoxylate as surfactant it is present in an amount of at least 1 wt.%, preferably from 1 wt.% to 10 wt.% and more preferably 1 wt.% to 6 wt.% of the composition.

The detergent composition of the invention generally contains a detergent surfactant in a range of from 5 to 60 wt.%, preferably 5 to 40 wt.% and most preferably from 10 to 25 wt.% of the composition.

According to the present invention the detergency builder system is based on zeolite MAP, optionally in conjunction with one or more supplementary builders. The amount of zeolite MAP employed may range, for example, from 5 to 60 wt.%, more preferably from 5 to 45 wt.%.

Zeolite MAP is described in EP 384070A (Unilever). It is defined as an alkali metal alumino-silicate of the zeolite P type having a silicon to aluminium ratio not greater than 1.33, preferably within the range from 0.9 to 1.33 and more preferably within the range of from 0.9 to 1.2.

Of particular interest is zeolite MAP having a silicon to aluminium ratio not greater than 1.15 and, more particularly, not greater than 1.07.

Zeolite P having a Si:Al ratio of 1.33 or less may be prepared by the following steps:

(i) mixing together a sodium aluminate having a mole ratio  $Na_2O:Al_2O_3$  within the range of from 1.4 to 2.0 and a sodium silicate having a mole ratio  $SiO_2:Na_2O$  within the range of from 0.8 to 3.4 with vigorous stirring at a temperature within the range of from 25 °C to boiling point usually 95 °C, to give a gel having the following composition;

 $Al_2O_3$ : (1.75-3.5)  $SiO_2$ : (2.3-7.5)  $Na_2O$ : P (80-450) $H_2O$ ;

- (ii) ageing the gel composition for 0.5 to 10 hours, preferably 2 to 5 hours, at a temperature within the range of from 70°C to boiling point, usually to 95°C, with sufficient stirring to maintain any solids present in suspension;
- (iii) separating the crystalline sodium aluminosilicate thus formed, washing to a pH within the range of from 10 to 12.5, and drying, preferably at a temperature not exceeding 150 °C, to a moisture content of not less than 5 wt.%.

Preferred drying methods are spray-drying and flash drying. It appears that oven drying at too high a temperature may adversely affect the calcium binding capacity of the product under certain circumstances.

Commercial sodium metasilicate pentahydrate dissolved in water and commercial sodium silicate solution (waterglass) are both suitable silica sources for the production of zeolite P in accordance with the invention. The reactants may be added together in any order either rapidly or slowly. Rapid addition at ambient temperature, and slow addition at elevated temperature (90-95 °C) both give the desired product.

Vigorous stirring of the gel during the addition of the reactants, and at least moderate stirring during the subsequent ageing step, however, appear to be essential for the formation of pure zeolite P. In the absence of stirring, various mixtures of crystalline and amorphous materials may be obtained.

Zeolite MAP generally has a calcium binding capacity of at least 150 mg CaO per g of anhydrous aluminosilcate, as measured by the standard method described in GB 1473201 (Henkel). The calcium binding capacity is normally 160 mg CaO/g and may be as high 170 mg CaO/g.

Although zeolite MAP like other zeolites contains water of hydration, for the purposes of the present invention amounts and percentages of zeolite are expressed in terms of the notional anhydrous material.

The amount of water present in hydrated zeolite MAP at ambient temperature and humidity is generally about 20 wt.%.

Preferred zeolite MAP for use in the present invention is finely divided and has a  $d_{50}$  (as defined hereinafter) within the range of from 0.1 to 5.0 micrometres. The quantity " $d_{50}$ " indicates that 50 wt.% of the particles have a diameter smaller than that figure.

A preferred zeolite MAP for use according to the present invention has a  $d_{50}$  of from 1.0 to 5.0 micrometres, for example 2.25 to 5 micrometres, more particularly 2.75 to 5 micrometres.

According to one embodiment of the invention the zeolite MAP detergent builder is in powder form.

For convenience in handling, however, the material may be granulated by conventional techniques such as spray drying or by a non-tower method to form larger particles.

The detergent composition according to the invention comprises soluble encapsulates of perfume.

The encapsulated perfumes comprise perfume dispersed in certain carrier materials.

In the context of this specification, the term "perfume" means any odoriferous material or any material which acts as a malodor counteractant. In general, such materials are characterised by a vapor pressure greater than atmospheric pressure at ambient temperatures. The perfume or deodorant materials employed herein will most often be liquid at ambient temperatures, but also can be solids such as the various tamphoraceous perfumes known in the art. A wide variety of chemicals are known for perfumery uses, including materials such as aldehydes, ketones, esters and the like. More commonly, naturally occurring plant and animal oils and exudates comprising complex mixtures of various chemical components are known for use as perfumes, and such materials can be used herein. The perfumes herein can be relatively simple in their composition or can comprise highly sophisticated, complex mixtures of natural and synthetic chemical components, all chosen to provide any desired odor.

Perfumes which are normally solid can also be employed in the present invention. These may be admixed with a liquefying agent such as a solvent prior to incorporation into the particles, or may be simply melted and incorporated, as long as the perfume would not sublime or decompose upon heating.

The invention also encompasses the use of materials which act as malodor counteractants. These materials, although termed "perfumes" hereinafter, may not themselves have a discernible odor but can conceal or reduce any unpleasant odors. Examples of suitable malodor counteractants are disclosed in U.S. Patent No. 3,102,101, issued August 27, 1963, to Hawley et al.

A wide variety of capsules exist which will allow for delivery of perfume effect at various times in the cleaning or conditioning process. The less protection provided results in greater perfume effect in product or washing/conditioning process. More protection results in greater perfume effect during the drying process or even later, after the surface has been treated.

Examples of such capsules with different encapsulated materials are capsules provided by microencapsulation. Here the perfume comprise a capsule core which is coated completely with a material which may be polymeric. U.S. Patent 4,145,184, Brain et al, issued March 20, 1979, and U.S. Patent 4,234,627, Schilling, issued November 18, 1980, teach using a tough coating material which essentially prohibits the diffusion out of the perfume. The perfume is delivered to fabric via the microcapsules and is then released by rupture of the microcapsules such as would occur with manipulation of the fabric.

Greater protection can be provided by choice of encapsulating material to be used to form the capsules, ratio of perfume to encapsulation or agglomeration of particles.

The choice of encapsulated material to be used in the perfume particles of the present invention will depend to some degree on the particular perfume to be used. Some perfumes will require a greater amount of protection than others and the encapsulating material to be used therewith can be chosen accordingly.

Nonlimiting examples of suitable water-soluble coating materials include such substances as methyl cellulose, maltodextrin and gelatin. Such coatings can comprise from about 1% to about 25% by weight of the particles.

Especially suitable water soluble encapsulating materials are capsules which consist of a matrix of polysaccharide and polyhydroxy compounds such as described in GB I,464,616.

Other suitable water soluble or water dispersible encapsulating materials comprise dextrins derived from ungelatinized starch acid-esters of substituted dicarboxylic acids such as described in US 3,455,838. These acid-ester dextrins are, preferably, prepared from such starches as waxy maize, waxy sorphum, sago, tapioca and potato. Suitable examples of said encapsulating materials are N-Lok®, manufactured by National Starch, Narlex ® (ST and ST2), and Capsul E ® . These encapsulating materials comprise pregelatinised waxy maize starch and, optionally, glucose. The starch is modified by adding monofunctional substituted groups such as octenyl succinic acid anhydride.

The perfume may also be encapsulated with a material that makes the particles more substantive to the surface being treated for example, fabric in the laundry process. Such materials help to deliver the particles

to the fabric and maximize perfume release directly on the fabric. Generally, these materials are water-insoluble cationic materials. Examples of useful material include any of the cationic (including imidazolinium) compounds listed in U.S. Patent 3,686,025, Morton, issued August 22, l972, incorporated herein by reference. Such materials are well known in the art and include, for example, the quaternary ammonium salts having at least one, preferably two,  $C_{10}C_{20}$  fatty alkyl substituent groups; alkyl imidazolinium salts wherein at least one alkyl group contains a  $C_8$ - $C_{25}$  carbon "chain"; the  $C_{12}$ - $C_{20}$  alkyl pyridinium salts, and the like.

Alternative materials useful for encapsulating materials to make them more fabric substantive are described in U.S. Patent 4,234,627, Schilling, issued November I8, I980, herein incorporated by reference.

The encapsulated perfume particles can be made by mixing the perfume with the encapsulating matrix by spray-drying emulsions containing the encapsulating material and the perfume. In addition, the particle size of the product from the spray-drying tower can be modified. These modifications can comprise specific processing steps such as post-tower agglomeration steps (e.g. fluidised bed) for enlarging the particle size and/or processing steps wherein the surface properties of the encapsulates are modified, e.g. dusting with hydrophobic silica in order to reduce the hygroscopicity of the encapsulates.

A particularly preferred encapsulation process is an emulsification process followed by spray-drying and finally dusting with silica. The emulsion is formed by:

- a) dispersing the starch matrix in water at room temperature in a I:2 ratio. It is preferred that the starch is pregelatinised so that the emulsion can be carried out at this temperature. This in turn minimises perfume loss. There must be a "low viscosity" starch to achieve high starch concentrations in water and high perfume loadings.
- b) the perfume oil is then added to the above mixture in the ratio of 0.8-1.05: I: 2, and the mixture is then emulsified using a high shear mixer. The shearing motion must produce oil droplets below I micron and the emulsion must be stable in this form for at least 20 mins (the function of the starch is to stabilise the emulsion once its mechanically made).
- c) the mixture is spray-dried in a co-current tower fitted with a spinning disk atomiser. The drying air inlet temperature is low I50-200 °C. This type of spray-drying ensures minimum loss of perfume and high drying rate. The granules have a particulate size of 50-I50 microns.
- d) the resulting dried encapsulates can contain up to 5% unencapsulated oil at the surface of the granules. To improve the flow characteristics up to 2% hydrophobic silica can be optionally added to the encapsulates via a ribbon blender.

It may be desirable to add additional perfume to the composition, as is, without protection via the capsules. Such perfume loading would allow for aesthetically pleasing fragrance of the composition itself. Upon opening the package containing the composition and as the product is added to water, this immediate release of fragrance may be desirable.

This perfume would be added via conventional means, e.g., mixing, as is, into a liquid composition or spraying onto dry product compositions.

In the granular detergent compositions according to the invention, the detergency builder can be zeolite MAP alone or a combination of zeolite MAP with an organic or inorganic cobuilder.

Suitable organic cobuilders can be monomeric or polymeric carboxylates such as citrates or polymers of acrylic, methacrylic and/or maleic acids in neutralised form. Suitable inorganic cobuilders include carbonates and amorphous and crystalline lamellar sodium silicates.

Suitable lamellar silicates have the composition:

## 45 NaMSi<sub>x</sub>O<sub>2x+1</sub>, yH<sub>2</sub>0

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where M is sodium or hydrogen, preferably sodium; x is a number from 1.9 to 4; and y is a number from 0 to 20. Such materials are described in US Patents No. 4664839; No. 4728443 and No. 4820439 (Hoechst AG). Especially preferred are compounds in which x = 2 and y = 0. The synthetic material is commercially available from Hoechst AG as S-Na<sub>2</sub> Si<sub>2</sub>O<sub>5</sub> (SKS6) and is described in US Patent No. 4664830.

The total amount of detergency builder in the granular composition ranges from 10 to 80 wt.%, more preferably from 15 to 60 wt% and most preferably from 10 to 45 wt.%.

Detergent compositions according to the invention may also suitably contain a bleach system. This preferably comprises one or more peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with bleach precursors to improve bleaching action at low temperatures.

The bleach system preferably comprises a peroxy bleach compound, preferably an inorganic persalt, optionally in conjunction with a precursor. Suitable persalts include sodium perborate monohydrolate and tetrahydrolate and sodium percarbonate, with sodium percarbonate being most preferred.

The Applicants have found that the present invention is particularly useful in detergent compositions comprising a percarbonate. Percarbonate is a poorer substrate to perfume than for example perborate and in conventional detergent compositions containing zeolite A as detergency builder, when perfume is sprayed on large perfume losses result on storage.

Percarbonate can be satisfactorily incorporated in the detergent compositions according to the invention employing zeolite MAP as detergency builder and using soluble capsules of perfume.

Preferred bleach precursors are peracetic acid precursors, such as tetraacetylethylene diamine (TAED); and peroxybenzoic acid precursors such as benzoyloxybenzene sulphonate (BOBS) and benzoyl caprolactam (BZCL).

The advantages afforded by the detergent composition of the present invention are particularly apparent in alkaline compositions, i.e. those which have a pH > 9.5 when measured on a 1% solution in distilled water. The composition according to the invention will preferably contain less than 6% by weight sulphate and less than 6% by weight bicarbonate.

Other materials which may be present in the detergent compositions of the invention include, for example, fluorescers, antiredeposition agents, inorganic salts such as sodium sulphate, enzymes, lather control agents, fabric softening agents, pigments, and coloured speckles.

The detergent compositions of the invention may be prepared by any suitable method. The particulate detergent compositions are suitably prepared by any tower (spray-drying) or non-tower process.

In processes based around a spray-drying tower, a base powder is first prepared by spray-drying a slurry and then other components, such as enzymes, unsuitable for processing via the slurry can be sprayed on or admixed (postdosed). The perfume capsules will generally be subsequently incorporated.

The zeolite MAP is suitable for inclusion in the slurry, although it may be advantageous for processing reasons for part of the zeolite MAP to be incorporated post-tower. The lamellar silicate, where this is employed, is also incorporated via a non-tower process and is preferably postdosed.

Alternatively, particulate detergent compositions in accordance with the invention may be prepared by wholly non-tower processes such as granulation.

The granular detergent compositions of the invention may be prepared to any suitable bulk density. The compositions should have a bulk density of at least preferably 400 g/l preferably at least 650 g/l, and, with particular preference at least 800 g/l.

The benefits of the present invention are particularly evident in powders of high bulk density, for example, of 700 g/l or above, which have a low porosity and hence are a poor substrate for sprayed-on perfume. Such powders may be prepared either by post-tower densification of spray-dried powder, or by wholly non-tower methods such as dry mixing and granulation; in both cases a high-speed mixer/granulator may advantageously be used. Processesusing high-speed mixer/granulators are disclosed, for example, in EP340 013A, EP 367 339A, EP 390 251A and EP 420 317A (Unilever).

The detergent composition according to the invention generally has a pH (as measured with a 1% solution in distilled water) of above 9.0, preferably above 9.5 and with particular preference about 10.

According to a further aspect, the invention provides use of a composition comprising zeolite MAP and soluble perfume capsules as an additive for a detergent composition.

Illustrative compositions according to the present invention are presented in the following Examples.

The following abbreviations are used in the Examples

45 AS : Sodium alkyl sulphate

LAS : linear  $C_{8-1\,1}$  alkyl benzene sulphonate.

TAED : Tetraacetyl ethylene diamine.

DTPMP : Diethylene triamine penta (methylene phosphonic acid), marketed by Mon-

santo under the Trade Name Dequest 2060.

50 MA/AA : Copolymer of 1:4 maleic/acrylic acid (M<sub>w</sub> 80,000).

Citrate : Trisodium citrate dihydrate.

CMC : Sodium carboxymethyl cellulose

SKS 6 : Lamellar sodium silicate (Hoechst AG).

TAE11 : tallow alcohol having an average of 11 ethylene oxide groups per mole

55 45AE7 : C<sub>14</sub>-C<sub>15</sub> alcohol ethoxylate having an average of 7 ethoxy groups per mole

of alcohol

AE3S : alcohol ethoxysulphate having an average of 3 ethoxy groups per mole.

silicone suds supressor : mixture of silanated silica : silicone in ratio of I:1.5 (M.wt.100,000)

# **EXAMPLES**

Particulate components and compositions were prepared as follows

5	Spray dried component	Α	В	С	D	Е
	Zeolite MAP MA/AA	13% 4%	13% 4%			10% I.82%
	DTPMP	0.5%	0.5%	-	-	0.2%
10	Brightener	0.2%	0.2%	-	-	-
	MgSO4	0.4%	0.4%	-	-	-
	Spray on					
15	Perfume	0.1%	-	-	-	-
	45 AE7	4%	4%	-	-	
	Silicone suds suppressor	0.5%	0.5%	-	-	
	TAE11	1%	1%	-	-	
	Post additives					
20	Percarbonate	18%	18%	18%	18%	
	TAED	5%	5%	5%	5%	
	SKS6	-	-	12%	12%	
	CITRATE	10%	10%	10%	10%	10%
25	Savinase (Trade Mark)					
	4.0 KNPU/g	1.5%	1.5%	1.5%	1.5%	1.5%
	Na Carbonate	8%	8%	12%	12%	20%
	Na Bicarbonate	2%	2%	2%	2%	5%
30	Surfactant agglomerate					
35	LAS	7%	7%			-
	C16/18AS	2%	2%	-	-	-
	C14/15AS	-	-	7%	7%	7%
	C12/15AE3 S	0.2%	0.2%	1.5%	1.5%	1.5%
	CMC	0.3%	0.3%	0.3%	0.3%	0.3%
	zeolite MAP	5%	5%	10%	10%	10%
	Na Carbonate	7%	7%	7%	7%	7%
	MA/AA	-	-	2%	2%	2%
	Perfume level (delivered in 40% active in dextrine capsules)	0.2%	0.3%	0.5%	0.3%	0.5%
40	Miscellaneous/Balance	100%	100%	100%	100%	100%
	Density	700g/l	700g/l	800g/l	800g/l	800g/l

# Claims

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- **1.** A granular detergent composition comprising:
  - (a) a surfactant selected from anionic, nonionic, cationic, amphoteric and zwitterionic detergent-active compounds and mixtures thereof;
  - (b) a detergency builder comprising zeolite P having a silicon to aluminium ratio not greater than 1.33 (zeolite MAP); and
  - (c) soluble encapsulates of perfume.
- 2. A granular detergent composition according to claim 1, which has a bulk density of at least 650g/l.
- 3. A granular detergent composition according to claim 2, which has a bulk density of at least 800g/l.
  - **4.** A granular detergent composition according to any one of claims 1 to 3 which comprises from 4 to 60% by weight of zeolite MAP.

- **5.** A granular detergent composition according to claim 4, which comprises from 4 to 45% by weight of zeolite MAP.
- **6.** A granular detergent composition according to any one of claims 1 to 5, wherein the zeolite MAP has a silicon to aluminium ratio not greater than 1.07.

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- 7. A granular detergent composition according to any one of claims 1 to 6, wherein the zeolite MAP has a particle size  $d_{50}$  of from 1.0 to 5.0 micrometres.
- 8. A granular detergent composition according to any one of claims 1 to 7, which contains percarbonate.
  - 9. A granular detergent composition according to any one of claims I to 8, which has a pH > 9.5 as measured on a I% solution in distilled water.
- **10.** A granular detergent composition according to any one of claims 1 to 9, wherein a part of the total perfume content is contained in soluble capsules and the remainder is sprayed on.
  - 11. A granular detergent composition according to any one of claims 1 to 10 which contains perfume encapsulated in soluble capsules in an amount of at least 0.1% by weight based on the total composition.

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