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(54) Detergent compositions.

(57) Particulate fabric washing detergent compositions are made with mixed phosphate detergency builders which comprise at least 5% of a water-soluble alkali metal polymer phosphate and at least 2% of an alkali metal orthophosphate in the ratio of 10:1 - 1:5 parts by weight, the total amount of these phosphates being from 10% to 40% of the composition and not more than 10% of other phosphate builders being present, together with 0.01% to 10% of an anti-deposition agent which is preferably an anionic polyelectrolyte.

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DETERGENT COMPOSITIONS

The present invention relates to powdered detergent compositions which are adapted for fabric washing, and which contain synthetic detergent active compounds together with mixed phosphate detergency builders. The invention concerns  
5 in particular the production of detergent compositions which contain levels of the mixed phosphate detergency builders which can be lower than conventional phosphate builder levels, but which still retain good detergency properties..

According to the present invention, a particulate  
10 detergent composition based on mixed phosphate detergency builders comprises a synthetic anionic, nonionic, amphoteric or zwitterionic detergent compound or mixture thereof, an alkali metal orthophosphate, an alkali metal or ammonium

polymer phosphate and an antideposition agent as defined below.

In UK patent No. 1,530,799 we described and claimed powdered alkaline fabric washing detergent compositions which essentially contain mixed alkali metal tripolyphosphate and alkali metal orthophosphate detergency builders in the ratio of from 10:1 to 1:5 parts by weight. These compositions were found to have surprisingly good detergency properties though containing lower levels of the phosphate detergency builders than in conventional sodium tripolyphosphate-based detergent compositions. This development enabled either a reduction in manufacturing cost because the difference in phosphate content could be made up with a cheaper filler, or an improvement in overall detergency by adding extra bleach or other additive instead. Additionally, this development facilitated the achievement of decreased phosphorus levels in detergent products to meet present or expected legislation.

Products of the type described in our aforementioned patent have already met with appreciable commercial success, especially as economy brands. However, one potential problem which has been found with the detergent compositions based on mixed alkali metal tripolyphosphate and orthophosphate builders, is the level of inorganic deposits which are sometimes found on washed fabrics and to a lesser extent surfaces in washing machines, especially heater elements. The present invention is directed to decreasing the level of such

deposits whilst retaining benefits of good detergency building properties.

Whilst not wishing to be bound by theory, it appears that the partial or complete replacement of the sodium tripolyphosphate in the detergent compositions of our  
5   afore-mentioned patent by the alkali metal or ammonium polymer phosphate gives less inorganic deposition on the washed fabrics, because the polymer phosphate has a decreased tendency to degrade to alkali metal pyrophosphate  
10   during the washing process. Also, by adding some and preferably all or at least a major part of the polymer phosphate to the detergent composition in discrete particulate form, as distinct from including the polymer phosphate in a spray dried base powder, the degradation of the polymer  
15   phosphate during spray drying which would form more alkali metal pyrophosphate can be avoided. We have found that the presence in the detergent composition of any such alkali metal pyrophosphate contributes particularly to inorganic deposits on the washing machines. Thus, by hindering the  
20   formation of alkali metal pyrophosphate during the washing process, and preferably also avoiding or substantially decreasing initial presence of the pyrophosphate in the detergent compositions, a major step forward is taken to control inorganic deposition on fabrics and washing machine  
25   surfaces better than hitherto, and yet still achieve good detergency building at relatively low phosphate levels.

In addition, the presence of an antideposition agent makes possible a further reduction in the level of inorganic deposition, especially on washed fabrics. It appears that the antideposition agents stabilise the  
5 suspended insoluble calcium orthophosphate particles, and hence inhibit their deposition on the fabrics. Thus, in accordance with the invention steps are taken to control the inorganic deposition caused by both precipitated calcium pyrophosphate and orthophosphate, giving substantial  
10 technical and hence commercial benefit.

The alkali metal polymer phosphate used in the compositions of the invention is preferably sodium polymer phosphate, but if desired the potassium or ammonium salts can be used. For convenience the term alkali metal polymer phosphate is used hereinafter and in the claims to include the  
15 sodium, potassium and ammonium salts. The alkali metal polymer phosphates are generally represented as having the formula  $M_2O(MPO_3)_n$ , where M is sodium, potassium or ammonium and n is an integer which is at least 4 and up to about 100  
20 or more, preferably from about 6 to about 50. In the case of the preferred sodium polymer phosphates, these figures correspond to a  $P_2O_5$  content of at least about 60.4% by weight up to a theoretical maximum of about 69.6%, preferably about 63% to about 69%  $P_2O_5$  by weight of the  
25 polymer phosphate. To be effective the polymer phosphates must of course be water-soluble. It should be appreciated that the chemical

structures of the polymer phosphates are not precisely defined, and some degree of ring formation or branching may be present in the otherwise normally linear polymer structure, especially with the shorter polymer chain lengths. The polymer phosphates are sometimes also known as glassy phosphates or Graham's salt.

It is preferred that all or at least a major part of the alkali metal polymer phosphate should be admixed with a detergent base powder, ie postdosed, to obtain the most benefit of lower alkali metal pyrophosphate content in the product. However, some or all of the alkali metal polymer phosphate can be included in the base powder if desired, and this can help to improve the powder properties, particularly when relatively low alkali metal orthophosphate levels are used. Any alkali metal polymer phosphate which is postdosed should, of course, have a suitable particulate form for postdosing, that is to say it should have an appropriate particle size range and powder density for uniform mixing with the base powder, so as to avoid undue segregation in the finished product.

Detergent compositions which are based on mixed phosphate builders and incorporate an alkali metal orthophosphate and discrete particles of alkali metal polymer phosphate, which can be made by postdosing the latter ingredient, are described in our co-pending UK patent application 39073/78 of even date.

The alkali metal orthophosphate used is either potassium or preferably sodium orthophosphate, as the latter is cheaper and more readily available. Normally the tri-alkali metal salts are used, but orthophosphoric acid or the di- or mono-alkali metal salts, e.g. disodium hydrogen orthophosphate or monosodium dihydrogen orthophosphate could be used if desired in the production of the compositions. In the latter event other more alkaline salts should also be present to maintain a high pH in the end product, i.e. with full neutralisation to the trialkali metal orthophosphate salts. The use of a mixture of the monosodium dihydrogen and disodium hydrogen orthophosphates in the ratio of 1:3 to 2:3, especially about 1:2, is particularly advantageous, as such a mixture (known as kiln-feed) is made in the production of sodium tripolyphosphate and is readily available. It is preferred to have all the alkali metal orthophosphate present in the detergent base powder, eg by inclusion in an aqueous detergent slurry and then spray dried, but part of the orthophosphate can be postdosed if desired, either separately or together with the alkali metal polymer phosphate.

The alkali metal orthophosphate can be used in the form of the anhydrous or hydrated salts, but in the former case it is preferred to promote hydration during processing, eg by adding the anhydrous orthophosphate to a detergent slurry and spray drying to form the base powder. The alkali metal polymer phosphates do not form hydrated salts as such, and

are normally used in anhydrous form, but they are hygroscopic and tend to absorb atmospheric moisture. The amounts of the salts used are expressed in anhydrous form.

The total amounts of the essential alkali metal polymer phosphate and alkali metal orthophosphate, and any other phosphates which may be present in the detergent compositions, are chosen according to the overall detergency builder level which is desired in the detergent compositions or according to the maximum permitted phosphorus content.

5 Normally the total phosphate builder level, which is preferably derived solely from alkali metal polymer phosphate and alkali metal orthophosphate, is between about 10% and about 40%, by weight of the composition, preferably with an amount of at least about 5% and at least about 2% up to about

15 20% each of the alkali metal polymer phosphate and orthophosphate, respectively. Preferably the amounts of the alkali metal polymer phosphate and alkali metal orthophosphate are each from about 5% to about 15%, especially about 5% to 10%, by weight of the composition. The total

20 amount of alkali metal polymer phosphate and alkali metal orthophosphate is preferably from about 10% to about 25%, especially about 15% to about 20%, by weight of the composition.

Generally speaking, it is preferred to have amounts of

25 the alkali metal polymer phosphate and the alkali metal orthophosphate generally within the ratio of from about 3:1 to about 1:3, especially about 2:1 to about 1:2, parts by

weight, respectively. These ratios of polymer phosphate to orthophosphate are particularly suitable for detergent compositions used at relatively high product concentrations, i.e. 0.3% to 0.8% by weight, as is common practice in Europe, especially in front-loading automatic washing machines, and where moderate levels of phosphates are allowed in the products, i.e. equivalent to 3% to 7% P.

It is preferable that the only phosphate detergency builders used to make the compositions of the invention should be the alkali metal polymer phosphate and alkali metal orthophosphate. In particular, it is desirable to add no alkali metal, ie sodium or potassium, pyrophosphates to the compositions as they tend to increase inorganic deposition as mentioned above. It is also preferred not to have any alkali metal tripolyphosphate present because of the relative ease with which it hydrolyses to form the alkali metal pyrophosphate. However, some alkali metal tripolyphosphate may be present if desired, either in the base powder or postdosed thereto, for example because of its beneficial effect on the base powder properties or because of its relative cheapness. But for optimum detergency in relation to total phosphate content, it is preferred to have not more than about 10% by weight of other alkali metal phosphate builders, and especially not more than about 5% of tripolyphosphate present in the detergent composition.

Some tripolyphosphate and pyrophosphate are also generally found as impurities at low levels in other

commercial alkali metal phosphates, and some tripolyphosphate and pyrophosphate may be formed by any hydrolysis of the polymer phosphate during processing, especially if some of it is present in the slurry rather than being all postdosed. Hence, total absence of alkali metal tripolyphosphate and pyrophosphate is generally unattainable in the detergent compositions. It is particularly preferred to have not more than about 2.5% of alkali metal pyrophosphate present in the compositions, as at higher levels the amounts of inorganic deposits on the washing machine parts become significantly more noticeable.

The detergent compositions of the invention essentially contain one or more antideposition agents to decrease the tendency to form inorganic deposits on washed fabrics. The antideposition agents appear to be effective by stabilising insoluble calcium orthophosphate particles in suspension. However, the best test for determining whether or not an antideposition agent is effective in a detergent composition according to the invention is to determine the level of inorganic deposits on fabric washed with and without the antideposition agent present. The most effective antideposition agents are generally anionic polyelectrolytes, especially polymeric aliphatic carboxylates. The amount of any such antideposition agent can be from about 0.01% to about 10% of the compositions, but is normally from about 0.1% to about 5% by weight, preferably from about 0.2% to about 2% by weight of the compositions.

Specific preferred antideposition agents are the alkali metal or ammonium, preferably the sodium, salts of homo- and co-polymers of acrylic acid or substituted acrylic acids, such as sodium polyacrylate, the sodium salt of copolymeth-  
5 acrylamide/acrylic acid and sodium poly-alpha-hydroxyacrylate, salts of copolymers of maleic anhydride with ethylene, acrylic acid, vinylmethylether or styrene, especially 1:1 copolymers, and optionally with partial esterification of the carboxyl groups. Such copolymers preferably have relatively low  
10 molecular weights, eg in the range of about 1,000 to 50,000. Other antideposition agents include the sodium salts of polymaleic acid, polyitaconic acid and polyaspartic acid, phosphate esters of ethoxylated aliphatic alcohols, polyethylene glycol phosphate esters, and certain organic phosphonic  
15 acids or salts thereof such as sodium ethane-1-hydroxy-1,1-diphosphonate, sodium ethylenediamine tetramethylene phosphonate, and sodium 2-phosphonobutane tricarboxylate. Mixtures of organic phosphonic acids or substituted acrylic acids or their salts with protective colloids such as gelatin  
20 may also be used. The most preferred anti-deposition agents are sodium polyacrylates having a MW of about 10,000 to 50,000, for example about 20,000 to 30,000.

The detergent compositions of the invention necessarily include from about 2.5% to about 50%, preferably about 5%  
25 to about 30%, and especially about 10% to about 25%, by weight of a synthetic anionic, nonionic, amphoteric or zwitterionic detergent compound or mixture thereof. Many

suitable detergent compounds are commercially available and are fully described in the literature, for example in "Surface Active Agents and Detergents", Volumes I and II, by Schwartz, Perry & Berch.

- 5       The preferred detergent compounds which can be used are synthetic anionic and nonionic compounds. The former are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms, the term alkyl being used to include
- 10      the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher ( $C_8-C_{18}$ ) alcohols produced for example from tallow or coconut oil; sodium and potassium alkyl ( $C_9-C_{20}$ )
- 15      benzene sulphonates, particularly sodium linear secondary alkyl ( $C_{10}-C_{15}$ ) benzene sulphonates; sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum; sodium coconut oil fatty
- 20      monoglyceride sulphates and sulphonates; sodium and potassium salts of sulphuric acid esters of higher ( $C_9-C_{18}$ ) fatty alcohol-alkylene oxide, particularly ethylene oxide, reaction products; the reaction products of fatty acids such as coconut fatty acids esterified with isethionic acid
- 25      and neutralised with sodium hydroxide; sodium and potassium salts of fatty acid amides of methyl taurine; alkane mono-sulphonates such as those derived by reacting alpha-olefins

(C<sub>8</sub>-C<sub>20</sub>) with sodium bisulphite and those derived by reacting paraffins with SO<sub>2</sub> and Cl<sub>2</sub> and then hydrolysing with a base to produce a random sulphonate; and olefin sulphonates, which term is used to describe the material made by reacting olefins, particularly C<sub>10</sub>-C<sub>20</sub> alpha-olefins, with SO<sub>3</sub> and then neutralising and hydrolysing the reaction product. The preferred anionic detergent compounds are sodium (C<sub>11</sub>-C<sub>15</sub>) alkyl benzene sulphonates and sodium (C<sub>16</sub>-C<sub>18</sub>) alkyl sulphates.

10        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

15        or with propylene oxide. Specific nonionic detergent compounds are alkyl (C<sub>6</sub>-C<sub>22</sub>) phenol-ethylene oxide condensates, generally 5 to 25 EO, ie 5 to 25 units of ethylene oxide per molecule, the condensation products of aliphatic (C<sub>8</sub>-C<sub>18</sub>) primary or secondary linear or branched

20        alcohols with ethylene oxide, generally 6 to 30 EO, or with both ethylene oxide and propylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called non-

25        ionic detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulfoxides.

Mixtures of detergent compounds, for example mixed anionic or mixed anionic and nonionic compounds may be used in the detergent compositions, particularly in the latter case to provide controlled low sudsing properties. This is  
5 beneficial for compositions intended for use in suds-intolerant automatic washing machines. We have also found that the use of some nonionic detergent compounds in the compositions decreases the tendency of insoluble phosphate salts to deposit on the washed fabrics, especially when used in  
10 admixture with some soaps as described below.

Amounts of amphoteric or zwitterionic detergent compounds can also be used in the compositions of the invention but this is not normally desired due to their relatively high cost. If any amphoteric or zwitterionic detergent compounds  
15 are used it is generally in small amounts in compositions based on the much more commonly used synthetic anionic and/or nonionic detergent compounds. For example, mixtures of amine oxides and ethoxylated nonionic detergent compounds can be used.

20 Soaps may also be present in the detergent compositions of the invention, but not as the sole detergent compounds. The soaps are particularly useful at low levels in binary and ternary mixtures, together with nonionic or mixed synthetic anionic and nonionic detergent compounds, which have low  
25 sudsing properties. The soaps which are used are the sodium, or less desirably potassium, salts of  $C_{10}-C_{24}$  fatty acids. It is particularly preferred that the soaps should

be based mainly on the longer chain fatty acids within this range, that is with at least half of the soaps having a carbon chain length of 16 or over. This is most conveniently accomplished by using soaps from natural sources such as tallow, palm oil or rapeseed oil, which can be hardened if desired, with lesser amounts of other shorter chain soaps, prepared from nut oils such as coconut oil or palm kernel oil. The amount of such soaps can be varied between about 0.5% and about 25% by weight, with lower amounts of about 0.5% to about 5% being generally sufficient for lather control. Amounts of soap between about 2% and about 20%, especially between about 5% and about 15%, can advantageously be used to give a beneficial effect on detergency.

Apart from the essential detergent compounds and detergency builders, the detergent compositions of the invention can contain any of the conventional additives in the amounts in which such materials are normally employed in fabric washing detergent compositions. Examples of these additives include lather boosters such as alkanolamides, particularly the monoethanolamides derived from palm kernel fatty acids and coconut fatty acids, lather depressants such as alkyl phosphates, waxes and silicones, antiredeposition agents such as sodium carboxymethylcellulose and polyvinyl pyrrolidone optionally copolymerised with vinyl acetate, oxygen-releasing bleaching agents such as sodium perborate and sodium percarbonate, per-acid bleach precursors, chlorine-releasing bleaching agents such as trichloroisocyan-

uric acid and alkali metal salts of dichloroisocyanuric acid, fabric softening agents, inorganic salts such as sodium sulphate, sodium carbonate and magnesium silicate, and, usually present in very minor amounts, fluorescent agents, perfumes, enzymes such as proteases and amylases, germicides and colourants.

It is particularly beneficial to include in the detergent compositions an amount of sodium perborate, preferably between about 10% and about 40%, for example about 15% to about 30%, by weight. It has been found that the bleaching action of sodium perborate is boosted under the highly alkaline conditions which also give optimum detergency building action for the alkali metal orthophosphate. Thus, it becomes possible to achieve improved bleaching properties by using the same levels of sodium perborate as normal; or decreased levels of sodium perborate can be used to give equal bleaching properties to those of conventional products with higher levels of perborate and sodium tripolyphosphate as the sole detergency builder. The latter option can also be used to further decrease the raw materials costs of the compositions, if a cheap filler is used in place of part of the sodium perborate.

It is also possible to include in the detergent compositions of the invention minor amounts, preferably not more than about 20% by weight, of other non-phosphate detergency builders, which may be either so-called precipitant builders, ion exchange or sequestrant builders. This is of

particular benefit where it is desired to increase detergency whilst using particularly low levels of the essential alkali metal polymer phosphate and alkali metal orthophosphate builders, so as to achieve especially low phosphorus contents in the detergent compositions. Examples of such other detergency builders are amine carboxylates such as sodium nitrilotriacetate, sodium carbonate, sodium aluminosilicate ion-exchange materials such as zeolites A and X, sodium citrate and soap, which can function as a detergency builder, as discussed above. However, such other builder materials are not essential and it is a particular benefit of using the mixed alkali metal polymer phosphate and orthophosphate that satisfactory detergency properties can be achieved at lower total phosphate levels than hitherto considered necessary without other detergency builders.

It is generally also desirable to include in the compositions an amount of an alkali metal silicate, particularly sodium ortho-, meta- or preferably neutral or alkaline silicate. The presence of such alkali metal silicates at levels of at least about 1%, and preferably from about 5% to about 15%, by weight of the compositions, is advantageous in decreasing the corrosion of metal parts in washing machines, besides giving processing benefits and generally improved powder properties. The more highly alkaline ortho- and meta-silicates would normally only be used at lower amounts within this range, in admixture with the neutral or alkaline silicates.

The compositions of the invention are required to be alkaline, but not too strongly alkaline as this could result in fabric damage and also be hazardous for domestic usage. In practice the compositions should give a pH of from 9 to 11 in use in aqueous wash solution. It is preferred in particular for domestic products to have a minimum pH of at least 9.25 and especially a pH of 9.5 or over, as lower pHs tend to be less effective for optimum detergency building, and a maximum pH of 10.5, as more highly alkaline products can be hazardous if misused. The pH is measured at the lowest normal usage concentration of 0.1% w/v of the product in water of 12<sup>0</sup>H (Ca), (French permanent hardness, calcium only) at 50<sup>0</sup>C so that a satisfactory degree of alkalinity can be assured in use at all normal product concentrations.

The pH of the detergent compositions in use is controlled by the amount of alkali metal orthophosphate and any other alkaline salts such as alkali metal silicate, sodium perborate and sodium carbonate present. The presence of such other alkaline salts, especially the alkali metal silicates, is particularly beneficial, because the alkalinity of the alkali metal orthophosphate is diminished in hard water due to precipitation of the calcium salt. In addition the alkali metal polymer phosphate is more stable and resistant to hydrolysis under highly alkaline conditions. The other ingredients in the alkaline detergent compositions of the invention should of course be chosen for

alkaline stability, especially the pH sensitive materials such as enzymes.

The detergent compositions of the invention are normally made in particulate form, for example by slurry making and spray drying, preferably with admixture of the alkali metal polymer phosphate to a spray dried base powder. However, if desired the detergent compositions may be compressed or compacted into tablets or blocks, or otherwise treated for example by granulation, prior to packaging and sale. Because of the hygroscopic nature of the alkali metal polymer phosphates, it may be desirable to use moisture impermeable packaging for the detergent compositions, for example in plastic or fabric sachets containing pre-measured doses for washing machine usage.

The invention is illustrated by the following Examples in which parts and percentages are by weight except where otherwise indicated.

Examples 1 to 9

A series of detergent powders were prepared by firstly spray drying a detergent base powder to the following formulation:

	<u>Ingredient</u>	% (based on final product)
	Sodium alkyl benzene sulphonate	9
	Nonionic detergent compound <sup>1</sup>	1
	Sodium orthophosphate	8
5	Sodium silicate (Na <sub>2</sub> O:SiO <sub>2</sub> , 1:2)	10
	Magnesium silicate	0.5
	Sodium sulphate	27.2
	Water and minor additives	9.3
		<hr/> 65.0

10      <sup>1</sup> C<sub>14</sub>-C<sub>15</sub> alcohol - 18 EO condensate.

Amounts of additives as specified in the Table below were then added to this base powder to make the formulations as follows:

	<u>Ingredient</u>	<u>Ex:</u>	<u>%</u>								
			<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>
15	Base powder	65	<hr/>								
	Sodium polymer phosphate <sup>2</sup>	10	-	-	10	-	-	10	-	-	-
	Sodium polymer phosphate <sup>3</sup>	-	10	-	-	10	-	-	10	-	-
	Sodium polymer phosphate <sup>4</sup>	-	-	10	-	-	10	-	-	10	-
20	Sodium polyacrylate <sup>5</sup>	-	-	-	2	2	2	1	1	1	-
	Sodium sulphate	2	2	2	-	-	-	1	1	1	-
	Sodium perborate	20	<hr/>								
	Nonionic detergent	2	<hr/>								

(continued overleaf)

(Continued)		<u>%</u>									
<u>Ingredient</u>	<u>Ex:</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	
Enzyme and perfume		0.4	—————→								
Lather controller <sup>6</sup>		0.6	—————→								
5       2	Polymer chain length 6-8.										
3	Polymer chain length 15-20 (plus 0.36% NaOH for pH control).										
4	Polymer chain length 25-30 (plus 0.72% NaOH for pH control).										
5	Versicol E7 (molecular weight 27000).										
6	Alkyl phosphate.										

10       These detergent compositions were evaluated for washing performance, including especially inorganic deposition on fabrics. The tests were accomplished in laundrometer machines using a product concentration of 8 g/l in water of 40° French hardness at 95°C. The levels of deposition on

15       the fabrics were determined after 10 repeat wash cycles using standard fabrics as indicated.

		<u>% inorganic deposition</u>									
<u>Fabric</u>	<u>Ex:</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	
Cotton poplin		0.6	0.5	0.7	0.2	0.2	0.2	0.3	0.1	0.2	
20   Polyester/cotton blend (50:50)		0.5	1.0	1.0	0.2	0.2	0.2	0.2	0.2	0.2	
Unsize cotton		1.8	2.0	2.1	0.6	0.3	0.2	0.8	0.3	0.3	

      These results are good in comparison with other reduced phosphate builder products, and show especially the benefit of using polyacrylate in conjunction with the orthophosphate-

25       polymer phosphate builder mixture. A comparative product

with 12% sodium tripolyphosphate instead of the polymer phosphate and with no polyacrylate gave 1.8%, 2.9% and 6.0% deposition on the same fabrics, respectively, under the same test conditions.

5 Examples 10 - 14

A series of detergent compositions were prepared by mixing the ingredients to the following formulation:

	<u>Ingredient</u>	<u>%</u>
	Sodium alkyl benzene sulphonate	6
10	Nonionic detergent compounds (mixed alcohol - 12 and 18 EO)	4
	Sodium polymer phosphate	6
	Sodium orthophosphate	6
	Sodium silicate	12
	Sodium carboxymethylcellulose	1
15	Sodium polyacrylate	2
	Sodium sulphate	33
	Sodium perborate	22
	Water and minor additives	to 100

The sodium polymer phosphate used had a chain length (n) of about 18 and the sodium polyacrylates used had molecular weights as follows:

	Ex. 10 - MW	4,000
	Ex. 11 - MW	18,000
	Ex. 12 - MW	30,000
25	Ex. 13 - MW	50,000
	Ex. 14 - MW	210,000

The compositions were then tested for detergency and inorganic deposition on washed fabrics (ash). Considering the low phosphate builder level the detergency properties were good, and the ash levels were particularly low, being  
5 between 0.5% and 0.9% on cotton poplin fabric and between 1.5% and 2.0% on Krefeld cotton after 10 wash cycles. The tests were conducted at a product concentration of 8 g/l in an Atlas Launderometer at 95°C in 23°GH water. By way of comparison, the ash levels for a similar sodium tripoly-  
10 phosphate-sodium orthophosphate built formulation were 2.0% and 5.7% respectively, under the same conditions.

Examples 15 - 18

Four detergent compositions were made to the formulation below:

15	<u>Ingredient</u>	<u>%</u>
	Sodium alkylbenzene sulphonate	7
	Nonionic detergent compounds (12-18 EO)	5
	Sodium orthophosphate <sup>1</sup>	6.0
	Sodium polymer phosphate <sup>1</sup> (chain length 18)	10.0
20	Sodium silicate	12.0
	Sodium sulphate	28.0
	Antideposition agent <sup>1,2</sup>	2.0
	Sodium perborate <sup>1</sup>	22
	Water and minor additives	to 100

25 <sup>1</sup>Added in particulate form to the dry-mixed base powder.

<sup>2</sup>In Example 15 - Sodium polyacrylate (MW 27,000)

- " " 16 - Ethylenediaminetetramethylene phosphonic acid
- " " 17 - Polyhydroxy acrylic acid
- " " 18 - Polymaleic acid (MW 1,400).

5           These compositions were evaluated for detergency and inorganic fabric deposition, and found to be good in both respects. The average figures for the latter on different cotton types after 10 wash cycles were only 0.3%, 0.65%, 0.75% and 1.3%, respectively, under the same conditions as  
10 for Examples 10 to 14.

Similar results were also achieved when sodium ethane-1-hydroxy-1,1-diphosphonate and other molecular weight polyacrylates were used as antideposition agents in the formulations.

## CLAIMS

1. A particulate alkaline detergent composition comprising from about 2.5% to about 50% by weight of a synthetic anionic, nonionic, amphoteric or zwitterionic detergent compound or mixture thereof and mixed phosphate detergency builders, characterised by incorporating at least about 5% by weight of a water-soluble alkali metal polymer phosphate having the formula  $M_2O-(MPO_3)_n$  wherein M is alkali metal and  $n$  is an integer of at least 4, and at least about 2% by weight of an alkali metal orthophosphate, the ratio of polymer phosphate to orthophosphate being about 10:1 to about 1:5 parts by weight, the total amount of the alkali metal polymer phosphate and alkali metal orthophosphate being from about 10% to about 40% by weight of the composition and not more than about 10% of other alkali metal phosphate builders being in the composition, and from about 0.01% to about 10% by weight of an anti-deposition agent which is an anionic polyelectrolyte.

2. A detergent composition according to claim 1, characterised that the alkali metal polymer phosphate is in sodium salt form.

3. A detergent composition according to claim 1 or claim 2, characterised that in the formula of the alkali metal polymer phosphate n is from about 6 to about 50.

4. A detergent composition according to any of the preceding claims, characterised that the amount of alkali metal polymer phosphate is from about 5% to about 15% by weight of the composition.

5. A detergent composition according to any of the preceding claims, characterised that the alkali metal orthophosphate is trisodium orthophosphate, disodium monohydrogen orthophosphate, monosodium dihydrogen orthophosphate or a mixture thereof.

6. A detergent composition according to any of the preceding claims, characterised that the amount of alkali metal orthophosphate is from about 5% to about 15% by weight of the composition.

7. A detergent composition according to any of the preceding claims, characterised that the total amount of the alkali metal polymer phosphate and of the alkali metal orthophosphate is from about 10% to about 25% by weight of the composition.

8. A detergent composition according to any of the preceding claims, characterised that the ratio by weight of alkali metal polymer phosphate to alkali metal orthophosphate is from about 3:1 to about 1:3.
9. A detergent composition according to any of the preceding claims characterised by incorporating not more than about 5% by weight of alkali metal tripolyphosphate.
10. A detergent composition according to any of the preceding claims, characterised by incorporating not more than about 2.5% by weight of alkali metal pyrophosphate.
11. A detergent composition according to any of the preceding claims, characterised that the anti-deposition agent is a polymeric aliphatic carboxylate.
12. A detergent composition according to any of claims 1 to 10, characterised that the antideposition agent is an organic phosphonic acid or a salt thereof.

c:

13. A detergent composition according to claim 11, characterised that the polymeric aliphatic carboxylate is sodium polyacrylate.

14. A detergent composition according to any of the preceding claims, characterised that the amount of the antideposition agent is from about 0.1% to about 5% by weight of the composition.

15. A detergent composition according to any of the preceding claims, characterised by the presence of from about 0.5% to about 25% by weight of soap.

16. A detergent composition according to any of the preceding claims, characterised by the presence of from about 10% to about 40% by weight of sodium perborate or percarbonate.



European Patent  
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# EUROPEAN SEARCH REPORT

0009954

Application number

EP 79 302 059.5

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. CL)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
X	<p><u>DE - A1 - 2 637 890 (UNILEVER)</u></p> <p>* claims 1 to 3, 7, 8, 12, 14, 21;</p> <p>page 23, paragraph 2, page 26, example 3, page 21, paragraph 1 *</p> <p>&amp; FR - A1 - 2 322 198</p> <p>--</p>	<p>1-7,</p> <p>9-11,</p> <p>12-16</p>	C 11 D 3/06
X	<p><u>DE - A1 - 2 606 684 (PROCTER &amp; GAMBLE)</u></p> <p>* claims 1 and 8 *</p> <p>--</p>	1-4	
P,X	<p><u>DE - A1 - 2 816 770 (PROCTER &amp; GAMBLE)</u></p> <p>* claims 1, 4 to 7, 12 and 23 *</p> <p>--</p>	<p>1-5,7,</p> <p>10,12,</p> <p>16</p>	<p>TECHNICAL FIELDS SEARCHED (Int. CL)</p>
A	<p><u>US - A - 4 116 852 (J. BAILEY et al.)</u></p> <p>* complete document *</p> <p>----</p>		C 11 D 3/00
			CATEGORY OF CITED DOCUMENTS
			<p>X: particularly relevant</p> <p>A: technological background</p> <p>O: non-written disclosure</p> <p>P: intermediate document</p> <p>T: theory or principle underlying the invention</p> <p>E: conflicting application</p> <p>D: document cited in the application</p> <p>L: citation for other reasons</p>
			&: member of the same patent family, corresponding document
X	The present search report has been drawn up for all claims		
Place of search		Date of completion of the search	Examiner
Berlin		10-12-1979	SCHULTZE